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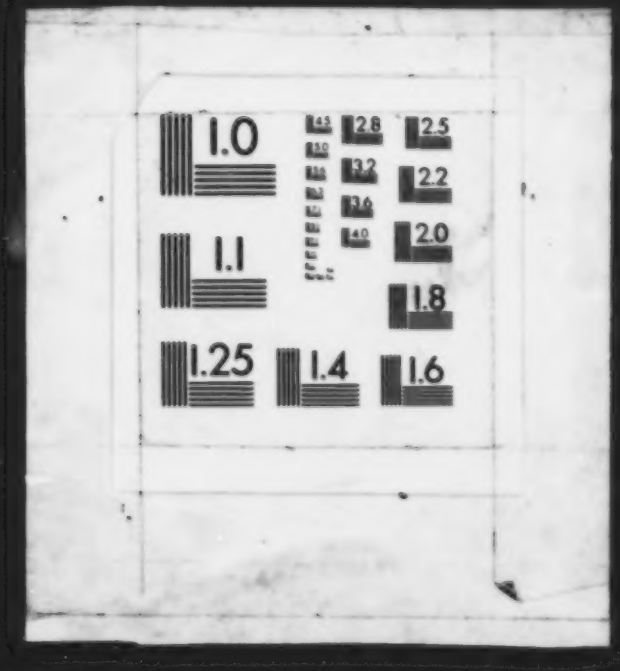

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PEI Associates, Inc., Cincinnati, OH

Prepared for

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ASSESSMENT OF ASSAY METHODS FOR EVALUATING ASBESTOS ABATEMENT TECHNOLOGY AT THE CORVALLIS ENVIRONMENTAL RESEARCH LABORATORY

by

PEI Associates, Inc.
Cincinnati, Ohio 45246-0100

Contract No. 68-03-3197

Project Officer

William C. Cain
Manufacturing and Service Industries Branch
Water Engineering Research Laboratory
Cincinnati, Ohio 45268

WATER ENGINEERING RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
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FOREWORD

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water systems. Under a mandate of national environmental laws, the agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. The Clean Water Act, the Safe Drinking Water Act, and the Toxic Substances Control Act are three of the major congressional laws that provide the framework for restoring and maintaining the integrity of our Nation's water, for preserving and enhancing the water we drink, and for protecting the environment from toxic substances. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Water Engineering Research Laboratory is that component of EPA's Research and Development program concerned with preventing, treating, and managing municipal and industrial wastewater discharges; establishing practices to control and remove contaminants from drinking water and to prevent its deterioration during storage and distribution; and assessing the nature and controllability of releases of toxic substances to the air, water, and land from manufacturing processes and subsequent product uses. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

This publication evaluates a particular aspect of assessing the nature and controllability of releases of toxic substances to the air. Specifically, it evaluates the sampling and analytical methods for determining the concentration of asbestos fibers in buildings that have undergone asbestos abatement. Aggressive and nonaggressive asbestos sampling methods are evaluated and compared, and the phase contrast and transmission electron microscopy analytical methods are evaluated and compared.

Francis T. Mayo, Director
Water Engineering Research Laboratory

ABSTRACT

Two methods were compared for analyzing the condition of a building after the removal of asbestos-containing materials: Phase contrast microscopy (PCM), the U.S. Environmental Protection Agency's (EPA's) current, nonaggressive sampling method, and transmission electron microscopy (TEM), an alternative aggressive sampling technique.

Air sampling was conducted at an EPA office building which had undergone an asbestos abatement program. The aggressive sampling technique revealed that air-entrainable asbestos remained in work areas after completion of abatement actions. The ratio of aggressive to nonaggressive PCM fiber concentrations was 7.0, whereas this ratio was 3.7 for TEM analyses. Study results also confirm that under similar sampling conditions, TEM analysis detects more fibers than PCM because of TEM's better resolving capability. The ratio of TEM/PCM concentrations for nonaggressive sampling was 3.0 for ambient samples and 3.3 for indoor samples; the ratio for aggressive sampling was about 2. Because the PCM method does not discriminate between asbestos and other fibers and cannot resolve fibers thinner than about 0.2 μm, PCM results may not accurately reflect the true hazard potential.

Study conclusions led to the following recommendations. Although time-consuming and expensive, TEM should be recommended as the analytical method of choice for measuring airborne asbestos fiber concentrations for final clearance testing of work areas after asbestos abatement. A criterion should be established that defines an acceptable asbestos fiber concentration in building areas after asbestos abatement, but not until a standardized TEM protocol and an aggressive sampling procedure are incorporated into asbestos guidelines. Continued research should focus on the development of a quicker, less expensive method for monitoring buildings after asbestos abatement and on more efficient abatement practices.

This report was submitted in fulfillment of Contract No. 68-03-3197 by PEI Associates, Inc., under the sponsorship of the EPA. This report covers a period from August 1984 to October 1985, and work was completed as of April 1986.

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Mr. Robert Amick served as Project Director and Mr. Mark Karaffa as Project Manager for PEI Associates. Ms. Ann Crone and Mr. Charles Zimmer assisted in the preparation of this report.

SECTION 1

INTRODUCTION

BACKGROUND

The Technical Assistance Program of the Office of Pesticides and Toxic Substances of the U.S. Environmental Protection Agency (EPA) provides guidance and information on the identification of asbestos-containing materials in buildings and on the correction of potential asbestos hazards. EPA Guidance Documents contain much of the technical information about asbestos in nonindustrial settings.[1-4] These documents describe how to establish an asbestos identification and control program, provide background information and direction to school officials and building owners on exposure assessment, and describe how to develop and implement an asbestos abatement program. The most recent asbestos guidance from EPA not only emphasizes recent experience and new information on asbestos control, but it also introduces and discusses criteria for developing an appropriate asbestos control plan.

Considerable scientific uncertainty still surrounds the technical considerations involved in assessing specific abatement actions to reduce the risk of asbestos exposure. One critical concern among the persons responsible for a program is how clean the asbestos-abatement contractor leaves a building (or building area) after removing the asbestos material or after completing work that could have disturbed an asbestos-containing material (e.g., encapsulation, enclosure, or special maintenance operations). The two criteria recommended in the version of EPA guidance (1983) that was in effect at the time of this study for evaluating the adequacy of the cleanup at the worksite are visual inspection of the worksite and air monitoring after completion of the project. Visual inspection should detect incomplete removal, any damage caused by abatement activity, and (most important) the presence of debris or dust that could contain asbestos as a result of inadequate cleanup of the work area. Air monitoring by the membrane filter collection technique and phase-contrast microscopic (PCM) analysis are recommended to supplement the visual inspection and to determine whether elevated levels of airborne fibers generated during the removal process have been sufficiently reduced. This currently recommended optical microscopic technique, one of two methods specified by the National Institute for Occupational Safety and Health (NIOSH) for determining airborne fiber concentrations, is used by the Occupational Safety and Health Administration (OSHA) for measurement of total airborne fibers in occupational environments.

The EPA-recommended air-monitoring methodology for determining abatement completion (NIOSH Method No. P&CAM 239) was as follows:

Air sampling should begin after the project has been completed and all surfaces in the abatement site have been cleaned, preferably within 48 hours after abatement work is finished. A minimum of three air monitors per worksite and at least one per room is recommended. Air is drawn through a membrane filter for about 8 hours at a flow rate of approximately 2 liters per minute. A total air volume of approximately 1,000 liters collected at the specified flow rate should be sampled. After the sampling, a section of the filter is mounted on a microscope slide and treated to form a transparent, optically homogenous gel. The fibers are sized and counted by using a phase-contrast microscope at 400 to 450X magnification. For counting purposes, a fiber is defined as a particle with a physical dimension longer than 5 micrometers and a length-to-diameter ratio of 3 to 1 or greater.[3]

This method is intended to give an index of the airborne concentration of asbestos fibers of specified dimensional characteristics in an atmosphere known or suspected to contain asbestos. It is not designed to count fibers less than 5 micrometers long or to differentiate asbestos fibers from other fibrous particulates.

The most significant limitation of the PCM method compared with the use of transmission electron microscopy (TEM) and scanning electron microscopy (SEM) is that the PCM method is limited in detecting fine particles (i.e., particles with submicron diameters, or lengths less than 5 μm) that are potentially toxicologically significant. For example, in glove-box tests of simulated industrial mechanical operations on asbestos-containing products (drilling, sawing, and sanding), the PCM methodology counted less than one percent of the fibers counted by TEM.[5] Although conditions of this glove box study are obviously different from asbestos-abatement activities, some concern existed about the relative merits and capabilities of the different analytical methods used to determine representative fiber concentrations. In another study, it was estimated that 50 to 100 times smaller asbestos fibers (i.e., fibers less than 0.2 μm wide and 5 μm long that are not detected by the PCM method) are present than the larger, optically visible fibers.[6]

The conditions in a work area during the time the final air samples are collected can influence the results of a post-abatement assessment. After an abatement action, the air is sampled while the area is sealed off, before ventilation is restored, and usually after at least a 24-hour settling period following the final wet cleaning. Consequently, this monitoring technique may not detect residual fibers that were missed by the cleaning or that have settled on horizontal surfaces during this static condition.

Residual asbestos fibers constitute a potential exposure hazard because they could be reentrained later when the air in the area is agitated by personnel traffic, air flow from ventilation systems, and custodial activities. Thus for more accurate characterization of postabatement fiber concentrations, the work area should have appreciable air movement, simulating actual use conditions while air monitoring is being conducted, to determine whether any reentrainable asbestos fibers remain in the area after completion of abatement activities. This introduction of air turbulence into the work area

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during the collection of stationary air samples is termed "aggressive sampling." This method entails the creation of air movement by the use of blowers, fans, brooms, or compressed air streams to entrain any particulate matter that may be present. Air sampling methods are further described in Section 5. The advantage of the aggressive sampling technique over the static (or nonaggressive) sampling is that the former reflects worst-case conditions and the testing requires a relatively short period. The disadvantages are that this technique is not readily standardized or reproducible, nor does it reflect normal exposure levels to occupants. As with the nonaggressive sampling method, no criteria have been established to define an acceptable or "safe" level of fibers in a nonoccupational environment.

"Guidance for Controlling Asbestos-Containing Materials in Buildings,"[4] issued by EPA in July 1985, recommends aggressive sampling and TEM analysis of air samples taken after an abatement action. These new guidelines contain a recommended protocol for aggressive sampling, a sampling strategy for postabatement clearance monitoring, and a statistical method for evaluating the TEM results and the adequacy of the contractor's cleanup.

OBJECTIVE

This study posed a problem-defining task designed to assess the adequacy of EPA's currently recommended optical microscopic method of analysis and sample collection technique compared with an electron microscopic method (TEM) and an alternative aggressive sampling technique. The results of this study will help to evaluate the advantages and/or technical limitations that could affect the application of the TEM analytical method and aggressive sampling technique in the assessment of air quality following asbestos abatement operations. In addition, the establishment of reliable methods of air sampling and analysis will permit the use of postabatement monitoring results to evaluate the efficacy of the methods for asbestos abatement and to develop better technical guidance for abatement contractors, building owners, and other parties directly responsible for remedial asbestos programs.

Because of the problem-defining nature of the study, the schedule and limited funding for this task did not allow for the development and implementation of a quality assurance project plan, which normally precedes such a field study. Active or recently completed abatement sites were selected for monitoring in this problem-defining study because they provided an excellent opportunity to collect real-world data and because the monitoring tasks could be arranged with minimum lead time and coordination.

REPORT ORGANIZATION

Section 2 of this report presents the study's conclusions, and Section 3 presents the recommendations. Section 4 describes the site selection criteria, sampling site, abatement program, and monitoring approach. Section 5 describes the nonaggressive and aggressive sampling procedures and the PCM and TEM methods used to analyze the filter samples collected at this site. Section 6 presents the air monitoring data, discusses their significance, and describes the statistical methods used for comparing the monitoring results.

SECTION 2

CONCLUSIONS

The following conclusions resulted from this study:

1. The aggressive sampling technique used in this problem-definition study revealed that air-entrainable asbestos fibers were present in previously abated areas. TEM analysis of aggressive samples from building areas that were never treated with asbestos insulation also revealed detectable levels of asbestos fibers. The levels found in the abated areas were not significantly higher than those in other areas of the building that had never been treated with asbestos material. These results suggest that 1) the abatement efforts had reduced asbestos fiber levels to an "equilibrium" level (i.e., fiber concentrations in the abated areas were the same as in other building areas), or 2) untreated building areas have been "contaminated" to some extent by asbestos from other areas via some transport mechanism.

2. Regardless of the analytical method used, the concentrations of fibers measured under aggressive sampling conditions were significantly higher than those measured under nonaggressive conditions. The ratios of aggressive to nonaggressive PCM fiber concentrations in abated and nonasbestos areas were 7.0 and 8.0, respectively, while these ratios by TEM analysis were 3.7 and 2.0. The PCM fiber concentrations during aggressive sampling conditions (0.002 to 0.057 fiber/cm^3) were well below the NIOSH-recommended occupational limit of 0.1 fiber/cm^3, an 8-hour, time-weighted average that is frequently cited in abatement contractor specifications as the final, post-abatement acceptance criterion. Alternatively, according to the 1983 EPA guidance document,[3] the lower detection limit was recommended as a standard for releasing the abatement contractor. The detection limit for a typical 1000-liter air sample analyzed by the NIOSH P&CAM 239 method would be about 0.03 fiber/cm^3. The most recent EPA guidance (1985) now recommends that all PCM sample analyses collected under aggressive conditions be less than 0.01 fiber/cm^3 and that the testing parameters be sufficient for reliable detection of such low levels. Thus, the PCM concentrations measured in the abated areas at this site during aggressive conditions exceed the EPA criteria for clearance monitoring. Under nonaggressive conditions, the PCM fiber concentrations in the abated work areas are all less than 0.01 fiber/cm^3 and thus would have met the former nonaggressive criterion.

3. The results of the study clearly demonstrate that, under similar sampling conditions, TEM analysis identifies more fibers than PCM. (The ratio of TEM/PCM fiber concentrations for nonaggressive sampling was 3.0 for ambient samples, 7.5 for indoor nonasbestos areas, and 3.3 for indoor abated samples; the ratios for aggressive sampling in indoor areas were about 2.) The PCM counting protocol specifies that only fibers 5 μm or longer are to be recorded. Because only fibers thicker than about 0.2 μm can be resolved by the light microscope, regardless of their lengths, thin fibers on the filter may not be detected or recorded by PCM. The TEM dimensional analysis reports on samples taken at the Corvallis site revealed that many of the asbestos fibers identified on the filters have widths less than 0.2 μm and lengths less than 5 μm.

   In addition, the PCM method does not discriminate between asbestos fibers and any other types of fibrous particulate. Thus, the value obtained from an environmental (nonoccupational) sample analyzed by only PCM may be totally unrelated to the presence or absence of any asbestos fibers. After an asbestos-removal project, any fibers left in the work area might reasonably be expected to be asbestos; however, it is not unusual for asbestos-containing building materials to contain other fibrous components, such as mineral wool, cellulose, or fibrous glass. Thus, the fibers detected by PCM after an abatement action are not always asbestos and may not accurately reflect the true hazard potential.

4. Concentrations of asbestos fibers in abated areas (as determined by TEM), measured by the aggressive sampling method, were significantly (6.2 times) higher than ambient TEM concentrations. The TEM concentrations under aggressive conditions in the nonasbestos areas were 5 times higher than ambient TEM concentrations; however, this difference was not statistically significant. The actual environmental conditions that exist in a building after reoccupancy, reactivation of ventilation systems, and the return to typical usage patterns are somewhere between the nonaggressive and aggressive sampling conditions. It is not likely that indoor conditions would ever be as rigorous as those created during the aggressive sampling conditions for this project. In addition, finishes applied during renovation of the building's interior subsequent to abatement (e.g., paint, carpeting, and suspended ceiling system), repeated cleanings, and continuous dilution of indoor air with ambient air would be expected to further reduce the possibility of residual fiber reentrainment and result in lower indoor concentrations. Over time, these concentrations would be expected to approach ambient levels. No data were obtained during this project to verify this theory.

In summary, this study, which was essentially completed before issuance of the 1985 EPA guidelines (Purple Book),[4] was designed to evaluate the methods of air sampling and analysis in the 1983 EPA guidance document.[3] The

conclusions presented in this report, which were based on actual air monitoring data from a large-scale asbestos-abatement project, support the recommendations for aggressive air sampling and TEM analysis for post-abatement air quality evaluations presented in the latest EPA guidance document.

SECTION 3

RECOMMENDATIONS

Based on the findings of this study, the following recommendations are made:

1. TEM should be recommended as the analytical method of choice for measuring airborne asbestos fiber concentrations for final clearance testing in atmospheres of buildings that have undergone asbestos abatement. The current TEM protocols, however, are very time-consuming and expensive for routine use in large abatement projects.

2. PCM analyses should be conducted as a preliminary check to determine whether additional cleanings are necessary before final clearance testing by TEM because PCM analyses are relatively inexpensive and can be performed quickly.

3. A criterion should be established that defines an acceptable asbestos fiber concentration in building areas after asbestos abatement, but not until a standardized TEM protocol and an aggressive sampling procedure have been developed and validated. Once developed, these methods should be required for all postabatement assessments.

4. Research should continue in the areas of asbestos measurement, sampling, hazard assessment, and abatement control technology so that asbestos hazards in buildings can be effectively reduced. One important research avenue should be the development of quicker, less expensive methods for monitoring the atmosphere in buildings after asbestos abatement.

SECTION 4

PROJECT DESCRIPTION

SITE SELECTION

Air monitoring was conducted at two sites where friable asbestos building materials had been removed:

Site 1. Columbus East High School
230 South Marr Road
Columbus, Indiana

Site 2. U.S. EPA Corvallis Environmental Research Laboratory
200 S.W. 35th Street
Corvallis, Oregon

This report describes only the results of the air monitoring survey conducted at Site 2. The monitoring data from Site 1 and the significance of these data are the subject of a separate report. The sites selected met the following criteria:

- The abatement plan involved the removal of friable, spray-applied, asbestos-containing material.
- The contractors carried out the work area preparation, removal, and decontamination in accordance with EPA-recommended specifications and requirements.[1]
- Multiple work areas containing homogeneous asbestos material were available for monitoring.
- The building owner and abatement contractor agreed to cooperate with EPA and to provide access to selected areas of the building.

The EPA Laboratory site was made available for this work as a result of collaborative agreement between the EPA Project Officer and the CERL Director, who had expressed interest in participating in this study.

BUILDING DESCRIPTION

The Corvallis Environmental Research Laboratory (CERL) is housed in a two-story, reinforced-concrete structure built in 1966. The building contains a total gross area of approximately 45,000 ft^2 (465 m^2). A single-pass HVAC system supplies the occupied building areas with 100 percent outside

air. The outside air enters through intakes on the roof, is tempered by heating or chilling coils, and is distributed by a closed-duct system to ceiling-mounted diffusers in all rooms and laboratories. Air flows through louvers in the bottom of interior doors and passes into the hallways (which serve as air plenums to the outside) or is exhausted through laboratory fume hoods. Figure 1 shows the floor plan of the main CERL facility.

Asbestos-containing Materials

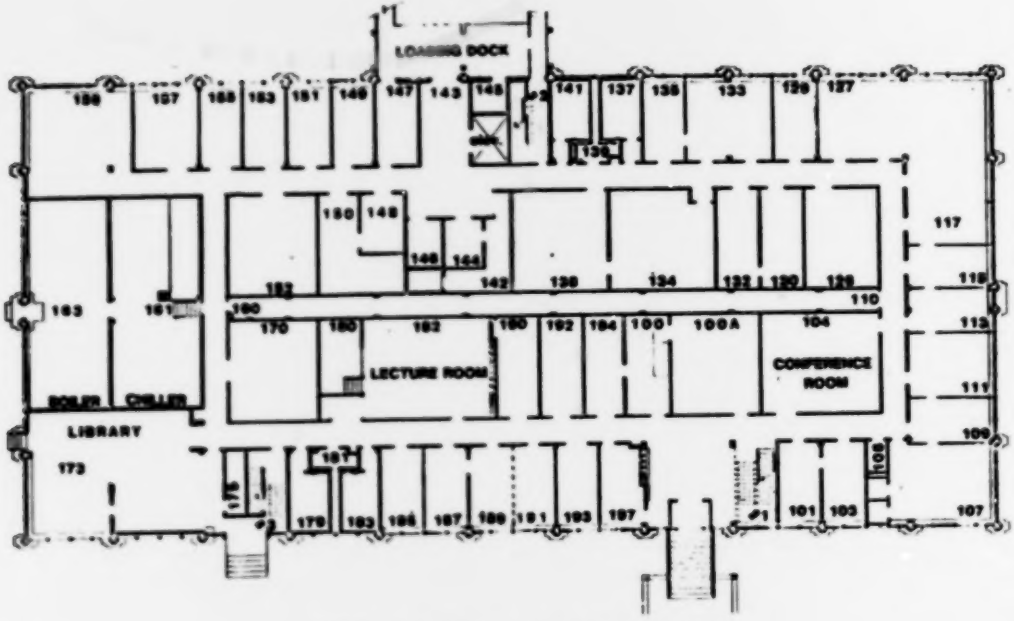
Asbestos-containing insulation had been spray-applied and tamped on the concrete ceilings (beams and deck) of four rooms (Rooms 146, 155, 157, and 159) and the penthouse in the main CERL facility and on beams in the boiler room (Room 163). The large air intakes located under the building, which supply ventilating air to the boiler and chiller room, were also lined with asbestos. The insulation material on the ceilings of Rooms 155, 157, and 159 and in the air ducts was removed in 1984 during a controlled abatement program. The asbestos-containing insulation in Room 146 (deionizer room), the boiler room, and the penthouse is still in place.

Samples collected from Room 146 and the penthouse were analyzed by polarized light microscopy and dispersion staining. The results indicated 80 percent amosite asbestos in each of the two bulk samples analyzed. At the time of the survey, the remaining insulation material was characterized as highly friable, loosely packed, and showing some signs of deterioration (loose, hanging pieces visible). Refer to Figures 2 and 3.

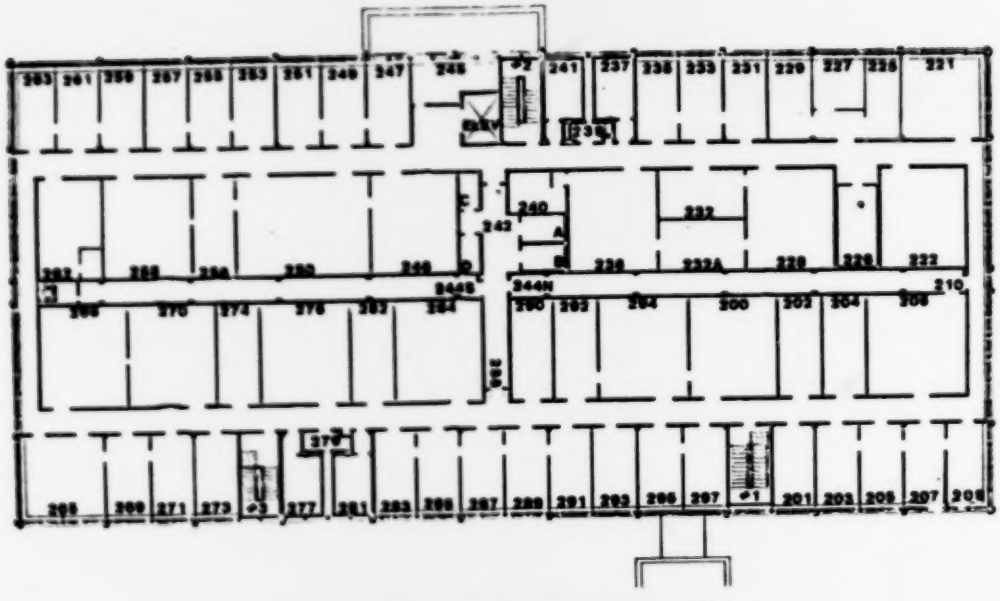
ABATEMENT PROGRAM

The asbestos-containing insulation in Rooms 155, 157, and 159 and in the air intakes was removed between May 21 and July 2, 1984. The abatement plan and schedule prepared by the contractor and submitted to CERL were reviewed and approved by EPA before work was begun. The work plan was in accordance with the EPA guidelines and EPA and OSHA asbestos regulations for asbestos removal and decontamination in effect at the time. Upon completion of the abatement effort, CERL personnel surveyed the work performed by the abatement contractor, performed additional cleaning of the work areas, and made arrangements for the painting of all ceiling surfaces from which the asbestos insulation had been removed.

According to CERL accounts, each work area was isolated from the rest of the building by temporary barriers. Ventilation ducts and openings to the outside or to adjacent rooms were sealed. Walls and floors were covered with plastic sheeting. Fully protected abatement workers first wetted the insulation with amended water and then scraped it off. The asbestos-containing debris was placed in sealable plastic bags and disposed of at a local EPA-approved sanitary landfill. Each work area was repeatedly wet-cleaned, and a settling period was allowed between cleanings. The ceiling surfaces were painted to bond any residual fibers that may have not been removed by the scraping, brushing, and wet-cleaning.



MAIN BUILDING, FIRST FLOOR



MAIN BUILDING, SECOND FLOOR

Figure 1. Floor plan of the main CERL building.

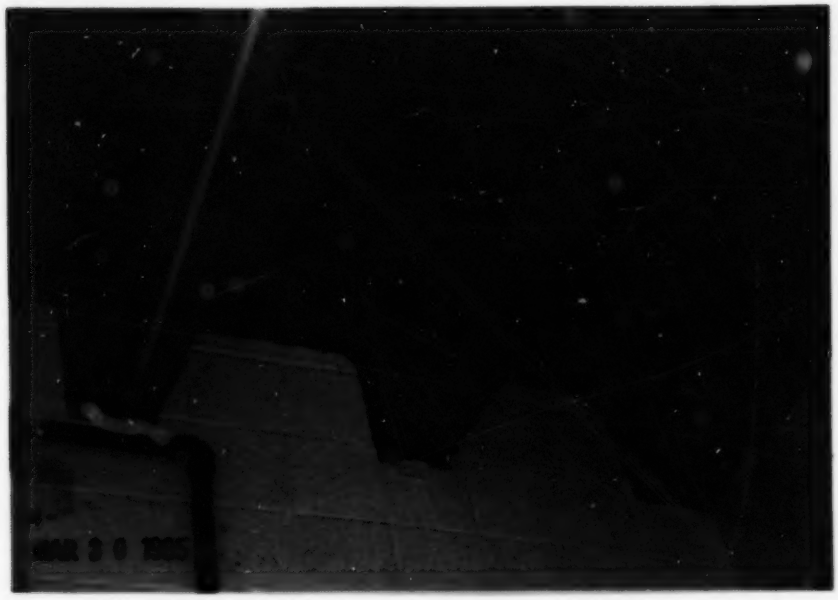


Figure 2. Asbestos-containing insulation on the ceiling of Room 146.

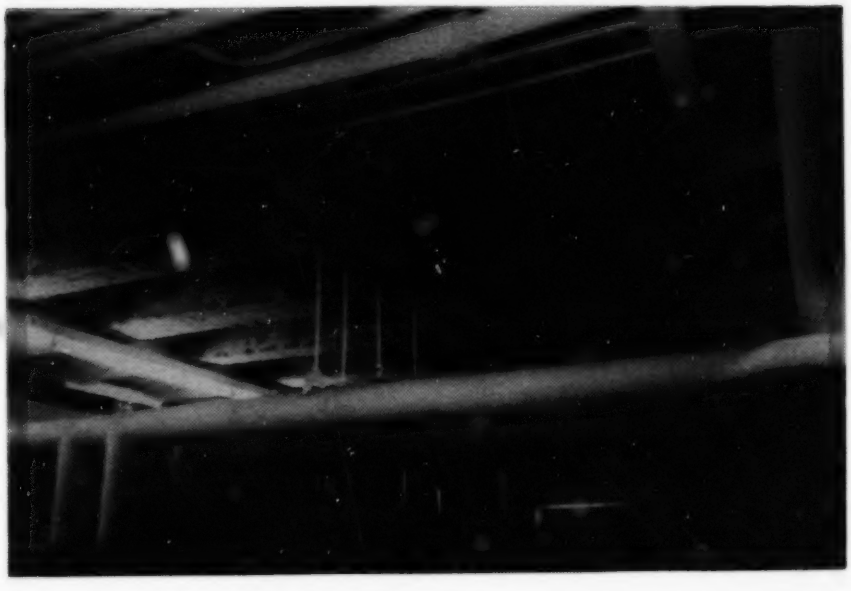


Figure 3. Asbestos-containing insulation on the ceiling of the penthouse.

50272-101

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15. Supplementary Notes: Original in French

16. Abstract (Limit: 200 words)

The National Institute of Applied Chemical Research (IRCHA) has developed a polyimide adhesive film for use at high temperatures equal to or more than 250°C. A pilot plant for making the film from a polyimide resin of French origin and transferring on to a glass fabric has been completed. Pictures of the film-making and transfer units along with some details of the process are included in the report. The film's adhesion and thermal resistance properties are superior to other adhesives of similar type. Longterm thermal stability studies are in process and would form the subject of a supplementary report. Figures available now show good stability at 250°C. Other physical properties and test results are given.

17. Document Analysis a. Descriptors

b. Identifiers/Open-Ended Terms: Foreign Technology

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MONITORING APPROACH

The sampling strategy for this study was to collect representative PCM and TEM samples 1) in rooms where friable asbestos-containing insulation had been removed, 2) in rooms that were never treated with asbestos-containing materials, and 3) outdoors. Samples for subsequent PCM and TEM analyses were collected from two or three representative locations in each room approximately 6 weeks after completion of all abatement activities. Two of the three monitored rooms from which asbestos insulation had been removed had been reoccupied. (Room 159 was vacant at the time of the survey.) Both nonaggressive and aggressive sampling techniques were used in each room. The nonaggressive or static sampling was conducted first, followed by the aggressive sampling. The nonaggressive sampling was performed on a weekday during regular working hours while the facility was in use and occupied. The aggressive sampling was conducted on a Saturday when the sampling areas were unoccupied. (The sampling procedures and analytical methods used in this study are described fully in Section 5 of this report.) To summarize briefly, filter holders containing either 0.8-μm Millipore mixed-cellulose ester (PCM) or 0.4-μm Nuclepore polycarbonate filters (TEM) were positioned 4 to 5 feet above the floor at arbitrary locations. Battery-powered sampling pumps were used to draw air through the filters. The constant-flow pumps were calibrated to 2.5 liters per minute and were operated for 8 hours per test. Samples were collected concurrently at outdoor locations during each monitoring period.

Upon completion of each monitoring survey, samples were submitted to the appropriate laboratory for preparation or analysis. The Nuclepore filters were carbon-coated before they were transported to the laboratory for TEM analysis.

SECTION 5

METHODS OF AIR SAMPLING AND ANALYSIS

OVERVIEW OF SAMPLING STRATEGY

Samples designated for PCM and TEM analysis were collected both aggressively and nonaggressively in six different rooms. Samples were also collected in the surrounding environment outside the building. The areas sampled included three rooms that had been treated previously with asbestos insulation and have since been abated, and rooms that have never been treated with asbestos. Representative PCM and TEM samples were collected approximately six weeks after all abatement activities had been completed. Outdoor air samples were collected on the roof of the building or at ground level in the open field west of the main building concurrently with the indoor samples.

Side-by-side (one PCM, one TEM) samples were collected in each room under nonaggressive and aggressive sampling conditions. The number of samples per room was not specified by study design; however, three of each type of sample were collected within each room. The nonaggressive sampling was performed first, during regular working hours with the building occupied and the HVAC system operating. The aggressive sampling was performed on a Saturday while the sampling areas were unoccupied and the HVAC system was not operating. Placement of the sampling equipment within each work area was the same during both nonaggressive and aggressive sampling.

SAMPLING METHODOLOGY

Sampling Equipment

Samples for subsequent PCM analysis were collected on 37-mm Millipore Type AA, mixed-cellulose ester membrane filters (0.8-μm pore size). The filters were preassembled by the manufacturer in three-stage polystyrene cassettes. Samples for TEM analysis were collected on 37-mm Nuclepore polycarbonate membrane filters (0.4-μm pore size). The polycarbonate membrane filter was supported within a three-stage polystyrene cassette by means of a support pad and backup filter (mixed-cellulose ester membrane, 5-μm pore size). Each sample cassette was sealed with a cellulose shrink band to prevent air from entering the sides of the unit during sampling.

Battery-operated personal sampling pumps equipped with rotameters and/or constant-flow controls were used to draw air through the sample filters. All sampling pumps were calibrated before and after sample collection with a

Magnehelic air flow gauge that had been calibrated by a soap-film flowmeter. The rotameter setting of each calibrated sampling pump was noted to provide a visual indication of proper pump functioning, and the settings were checked periodically throughout the sampling period.

Sample Collection and Handling

Samples designated for both PCM and TEM analyses were collected at a known flow rate of approximately 2.5 liters per minute (LPM). Sampling duration was 8 hours. The average sample volume per filter was 1200 liters.

All samples were collected open-faced (i.e., with the face cap of the cassette device removed) to expose the entire surface area of the filter. During sampling, the face caps were carefully stored in clean, resealable, plastic bags. The filter cassettes were positioned at breathing zone height [4.5 to 5.5 feet (1.4 to 1.7 m) above the floor] and were supported by taping the end of the sampling hose to the wall or clipping it to an adjustable tripod. The sample cassettes were also positioned so that the faces of the membrane filters were angled slightly toward the floor. Figure 4 shows a typical sampling apparatus.

At the end of the sampling period, each filter cassette was turned upright (i.e., the filter plane was parallel to the floor), the sampling pump was turned off, the face cap of the three-stage filter cassette was repositioned tightly on the cassette, the cassette was disconnected from the sampling hose, a plastic plug was inserted into the cassette outlet, and the cassette was placed face-up in a box for transport. All PCM and TEM filter samples were maintained in this upright position from the time of collection until they were carbon-coated or were analyzed by the appropriate laboratory. The PCM filters were hand-carried to the PEI Laboratory in Cincinnati, where they were subsequently analyzed.

The TEM samples were carbon-coated at the Electron Microscope Facility at Oregon State University in Corvallis and hand-carried to EPA in Cincinnati. The TEM samples were later shipped via overnight courier or hand-carried to the TEM laboratory in Chicago for analysis.

Nonaggressive Sampling

Samples for PCM and TEM analyses were collected under nonaggressive conditions for comparison with similar samples collected under aggressive conditions. The sampling condition was considered nonaggressive when air movement in the work area was negligible and/or minimized to the greatest possible extent. It is postulated that under this condition asbestos fibers (or any other particulate matter) will "settle out" if given sufficient time. The probability of reentrainment of these asbestos fibers is much lower during nonaggressive conditions than during conditions of typical building use or aggressive sampling conditions. In this study, nonaggressive sampling conditions existed when the work area was sealed off, all ventilation was

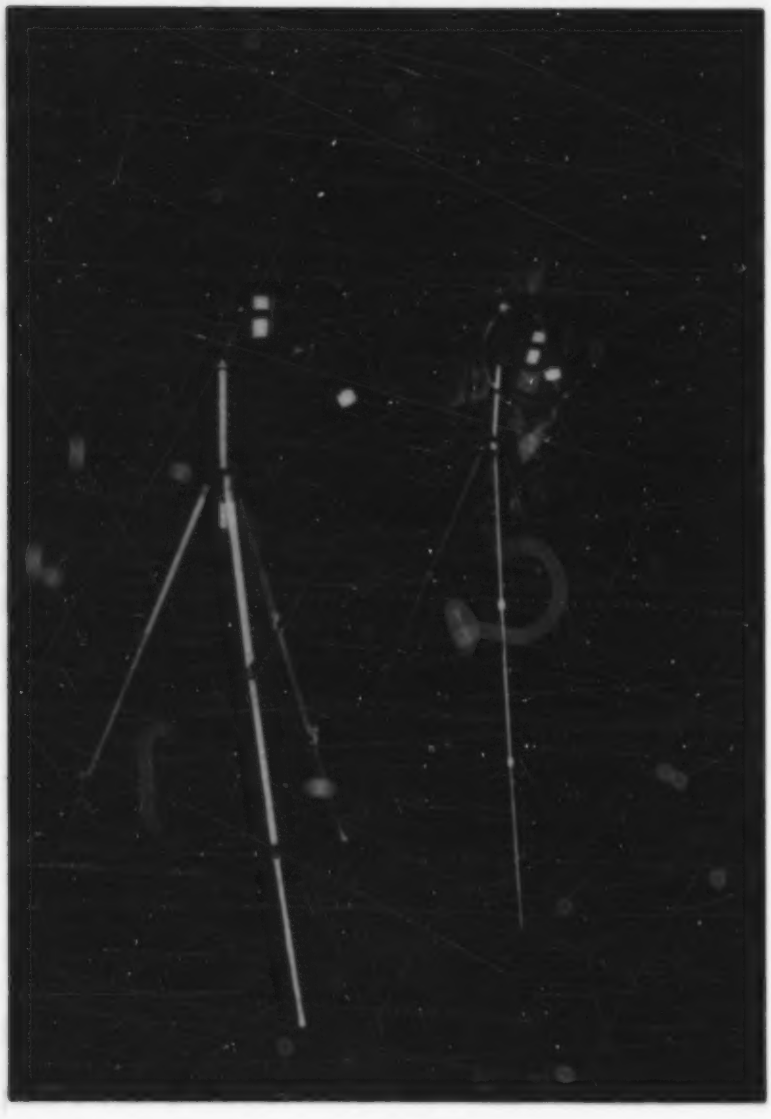

Figure 4. Photograph of a typical sampling apparatus. Personal sampling pump and filter cassette are positioned on an adjustable tripod.

shut off, and personnel access was prohibited. These are the typical conditions under which air monitoring is conducted at a work site following asbestos removal and decontamination.

Aggressive Sampling

Samples for PCM and TEM analyses were also collected under aggressive sampling conditions. Aggressive conditions were created by introducing air turbulence into the sampling area by intermittent use of a hand-held electric blower. The air movement created was much greater than would exist under conditions of normal building use. Under these aggressive sampling conditions, it is postulated that most asbestos fibers susceptible to entrainment would become airborne and remain suspended for the duration of the sampling period, as long as the fans were operated or the hourly introduction of air turbulence was continued. Thus, an aggressive environment provided the best possible setting for high or "worst-case" airborne asbestos fiber concentrations following abatement.

The blower used in this study was a 1-hp, electric power blower, as shown in Figure 5 and in the background in Figure 4. The airflow rate at the blower outlet is approximately 300 ft^3/min (8.5 m^3/min). The electric blower was equipped with a two-piece plastic tube extension and concentrator nozzle that enabled the operator to direct the airstream at objects and surfaces within the sampling area.

Aggressive sampling conditions were created in each of the work areas sampled by an initial "blow-down" of all surfaces, followed by hourly agitation with the blower throughout the duration of the sampling period. During aggressive sampling, the doors of the rooms were closed and building air handling systems were shut off. Figure 6 shows photographs of an aggressive sampling procedure in progress. The sequence of operations is summarized below.

1. A technician wearing full-body protective clothing and respiratory protection entered the work area, positioned the sampling equipment, and started the sampling pumps.

2. Using a back-and-forth motion, the technician directed the airstream of the electric blower at all surfaces within the sampling area (walls; floors; ceilings; all junctures between walls, ceilings, and floors; and any other exposed surfaces within the area enclosure). The technician then exited the sampling area.

3. After an elapsed time of approximately 1 hour, the technician reentered the room and directed the air steam from the blower back and forth across the floor for about a minute. This procedure was repeated hourly for the duration of the sampling period. Unless actively engaged in manipulating the electric blower or checking the sampling apparatus, the technician did not remain the room.

Figure 5. Photograph of the electric power blower used for aggressive sampling.

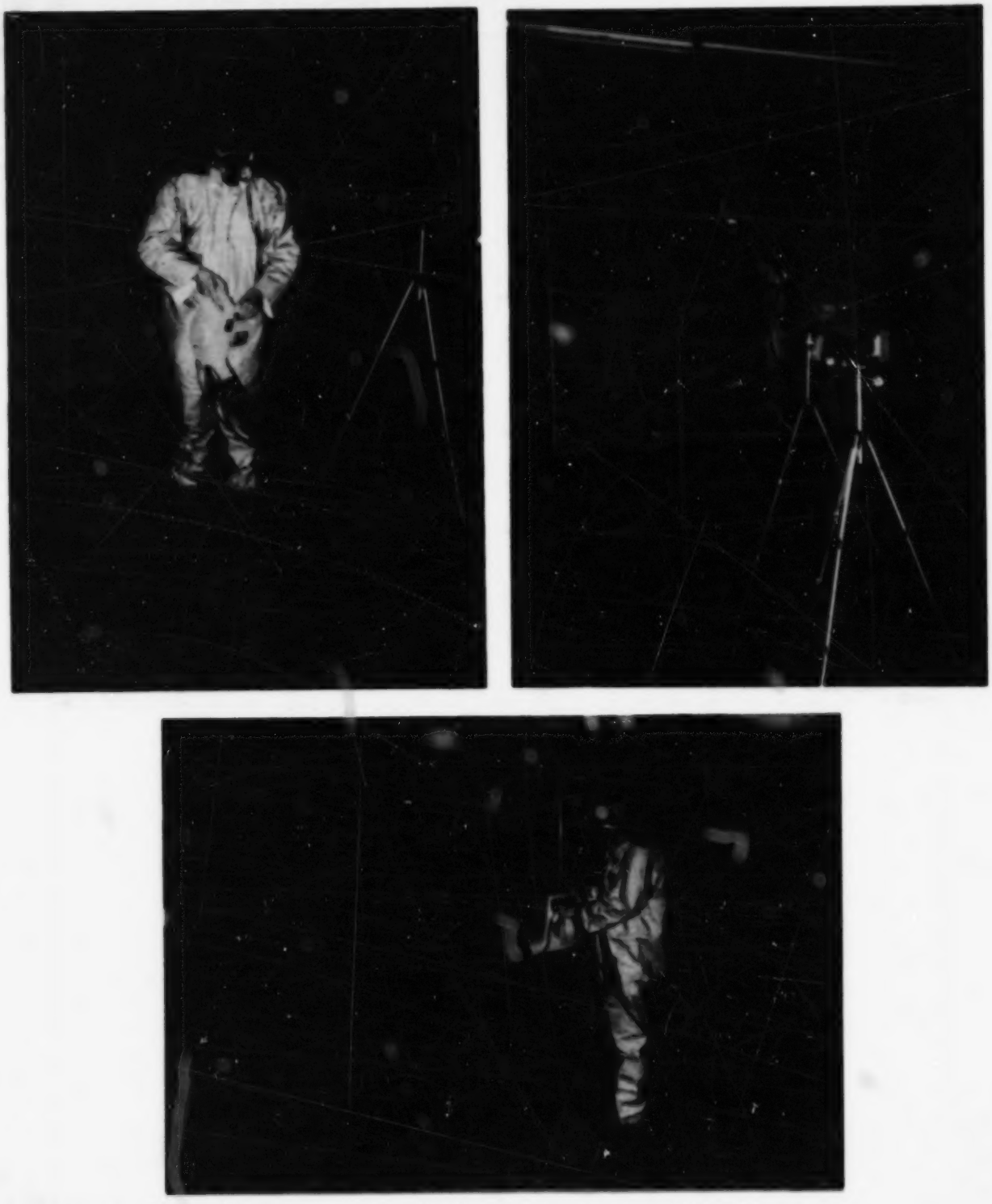

Figure 6. Photographs showing aggressive sampling in progress.

4. At the end of the sampling period, samples were collected, sampling pumps were turned off, and the sampling equipment was removed from the area.

The technician used appropriate respiratory protection and followed appropriate decontamination procedures.

METHODS OF ANALYSIS

Phase-Contrast Microscopy

All PCM samples were analyzed in accordance with NIOSH Method No. P&CAM 239.[7] This optical microscopic technique is the method the Occupational Safety and Health Administration uses to measure total airborne fibers in occupational environments. The 1983 EPA guidance document pertaining to asbestos in buildings (which was current at the time this study was conducted) recommends a visual inspection of the worksite, followed by air monitoring by the membrane-filter collection technique and phase-contrast microscopic analysis, as one method for evaluating satisfactory completion of asbestos abatement and decontamination of the worksite.[3]

Airborne fiber concentrations were determined by NIOSH Method No. P&CAM 239 through microscopic examination of the fibers collected on a mixed-cellulose ester membrane filter. A triangular wedge comprising approximately one-eighth of the entire surface area of the 37-mm-diameter filter was removed from the sample cassette, mounted on a microscope slide, and examined. The filter wedge was rendered into an optically transparent homogeneous gel by the use of a slide-mounting solution of 1:1 (by volume) dimethyl phthalate and diethyl oxalate. A microscope equipped with a phase-contrast condenser was used to size and count the fibers at 400-450X magnification. Only those fibers longer than 5 micrometers and having a length-to-width ratio of 3 to 1 or greater were counted. Fibers were sized by comparing fiber length with the diameters of the calibrated circles of a Porton reticle. Sample analysis continued until at least 20 fibers or 100 microscopic fields had been counted. Microscopic field areas generally range from 0.003 to 0.006 mm^2. The fiber-counting procedure followed the rules specified in the analytical method.

The estimated average airborne asbestos fiber concentration in the filter sample is calculated by using the following formula:

$$AC = \frac{[(FB/FL) - (BFB/BFL)](ECA)}{(FR)(T)(MFA)}$$

where

AC = Airborne fiber concentration in (fibers > 5 μm)/m^3
BFB = Total number of fibers counted in the BFL fields of the blank or control filters in fibers > 5 μm
BFL = Total number of fields counted on the blank or control filters

ECA = Effective collecting area of filter (855 mm^2 for a 37-mm filter with an effective diameter of 33 mm)
FR = Pump flow rate in liters/min (LPM)
FB = Total number of fibers counted in the FL fields in fibers > 5 μm
FL = Total number of fields counted on the filter
MFA = Microscope count field area in mm^2 (a field area 0.003136 mm^2 was used by the PEI Laboratory)
T = Sample collection time in minutes

The minimum total fiber count in 100 fields that is considered adequate for reliable quantitation is 10 fibers. Thus, the lower limit of reliable quantitation for this method is approximately 22,720 fibers/m^3 (or 0.023 fibers/cm^3 when 1200 liters of air is sampled). During this study, all of the PCM samples collected under nonaggressive conditions and several of the PCM samples collected under aggressive conditions yielded fiber counts less than the reliable quantitation limit (i.e., less than 10 fibers in 100 fields). The fiber concentrations of these samples were calculated and reported based on the actual number of asbestos fibers counted rather than merely "less than the limit of reliable quantitation" because it was believed this would provide valuable information about these data that otherwise would have been lost. The precision, accuracy, and coefficient of variation associated with sample results below the reliable level of quantitation have not been determined.

Analyses of several other PCM samples collected during this study yielded counts of zero fibers per 100 fields. Because one-half of one fiber is the smallest quantity permitted to be counted in the counting rules specified in P&CAM 239, these sample concentrations are reported as less than the lowest limit of detection (i.e., the fiber concentration based on counting 1/2 of a fiber in 100 fields) as shown in the following calculations:

$$\text{Detection limit} = \frac{\text{Number of fibers counted/100 fields}}{\text{Volume of air sampled } (m^3)} \times \frac{\text{Effective collecting area of the filter } (mm^2)}{\text{Microscopic field area } (mm^2/\text{field})}$$

$$\text{Sample calculation: } DL = \frac{0.5 \text{ fiber/100 fields}}{1.272\ m^3} \times \frac{855\ mm^2/\text{filter}}{0.003136\ mm^2/\text{field}} = 1072 \text{ fibers/m}^3$$

Transmission Electron Microscopy

Nuclepore filters were prepared and analyzed for asbestos content by TEM in accordance with the Methodology for the Measurement of Airborne Asbestos by Electron Microscopy by Yamate, et al.[8] The current TEM methodology was developed particularly for application to samples collected from a volume of

air in which the asbestos concentration is considered a minor component of the total particulate loading. Carbon-coating of the samples was performed by the staff of the Electron Microscope Facility at Oregon State University. Completion of sample preparation and sample analyses were performed by the TEM laboratory in Chicago.

Three levels of TEM analysis are described in the methodology. Briefly summarized, Level I TEM analysis involves examination of the particulates deposited on the sample filter by a 100-kV transmission electron microscope. Asbestos structures (fibers, bundles, clusters, and matrices) are counted, sized, and identified as to asbestos type (chrysotile, amphibole, ambiguous, or no identity) by morphology and by observing the selected area electron diffraction (SAED) patterns. The width-to-length ratio of each particle that is counted is recorded. Level II TEM analysis consists of a Level I analysis plus chemical elemental identification by energy-dispersive spectrum (EDS) analysis. Energy-dispersive analysis is used to determine the spectrum of the X-rays generated by an asbestos structure. X-ray elemental analysis is used for further categorization of the amphibole fibers, identification of the ambiguous fibers, and confirmation or validation of chrysotile fibers. All Nuclepore samples collected in this study were analyzed by Level II TEM. Level III TEM analysis (not used in this study) consists of a Level II analysis plus quantitative SAED of individual fibers. Quantitative SAED is a more extensive SAED analysis than that used for Level II. Fibers are examined from different orientations or viewing angles and compared with SAED patterns from asbestos mineral standards in Level III quantitative SAED.

Shortly after sampling was completed, the Nuclepore polycarbonate filters were carbon-coated. Carbon-coating, the first step in the sample preparation procedure, is accomplished by removing the face cap from the cassette holder to expose the surface area of the sample filter and then securing the open-faced holder to a rotating turntable for carbon-coating within a high-vacuum carbon evaporator. Once carbon-coated, a 3-mm-diameter section of the polycarbonate filter (usually midway between the center and edge) is placed on a 3-mm-diameter electron microscope grid. The polycarbonate membrane is then dissolved with solvent, which results in a membrane-free EM grid with particles embedded in the carbon film coating. An optional step in sample preparation is gold-coating (accomplished in a manner similar to that for carbon-coating). The thin gold coating provides an internal standard for SAED analysis.

The prepared samples were examined with the transmission electron microscope at 20,000X magnification for particulate counting and sizing. A minimum of 100 fibrous structures or 20 grid openings, whichever came first, were examined. (Analytical protocol requires that a minimum of 100 fibrous structures or 10 grid openings be examined. In this study, 20 grid openings were examined to lower the detection limit because very low fiber concentrations were expected in these postabatement samples.) The exact counting rules and sizing techniques are described in greater detail in Appendix A. In addition to particulate counting and sizing, the SAED patterns from all fibrous structures identified were observed. From visual examination of the

SAED pattern, a fibrous structure can be classified as belonging to one of four categories: 1) chrysotile, 2) amphibole group (includes amosite, crocidolite, anthophyllite, tremolite, and actinolite), 3) ambiguous, or 4) no identification. The SAED patterns cannot be identified for all particulates (particularly matrices/debris, clusters/clumps) because of the absence of a recognizable diffraction pattern. X-ray elemental analysis with EDS was used to categorize the amphibole fibers, to identify the ambiguous fibers, and to confirm or validate chrysotile fibers. A sample laboratory summary analysis report is shown in Figure 7. The dimensional analysis and EDS results for this sample are presented in Appendix B.

The fiber concentration of the filter sample is calculated by using the following equation:

$$\text{Fibers/m}^3 = \frac{\text{Total No. of fibers}}{\text{No. of EM fields}} \times \frac{\text{Total effective filter area, cm}^2}{\text{Area of an EM field, cm}^2} \times \frac{1}{\text{Volume of air sampled, m}^3}$$

The total effective filter area is 8.6 cm^2. The areas of the grid openings varied, typically ranging from 0.00005 to 0.00007 cm^2. The average grid opening area per sample was calculated and recorded on the laboratory analysis report.

The theoretical limit of detection for the TEM analyses performed was based on counting one fiber or structure in 20 grid openings. This limit of detection is calculated by the following formula:

$$\text{Detection limit} = \frac{1 \text{ fiber}}{\text{No. of grid openings scanned}} \times \frac{\text{Area of filter (cm}^2\text{)}}{\text{Area of grid opening (cm}^2\text{)}} \times \frac{1}{\text{Volume of air (m}^3\text{)}} = \frac{1 \text{ fiber}}{20} \times \frac{8.6 \text{ cm}^2}{0.63 \times 10^{-4} \text{ cm}^2} \times \frac{1}{1.2 \text{ m}^3} = 5688 \text{ fibers/m}^3$$

Analyses of two TEM samples collected during this study yielded counts of zero fibers or structures per 20 grid openings. These samples are reported at less than the detectable limit (as calculated by the aforementioned equation).

TEM Asbestos Analysis Report

Sample I.D.: COR-N31 05-170 Date Analyzed: 7/24/85 IITRI Sample No.: C06610-0120

Date Sample Received: 4/11/85 Sample Type: Bulk (Air) Water Misc. (circle one)

Filter Type: 37 mm Nuclepore Filtration Area (cm^2) 8.6 Volume of Fluid Sampled: 1150 L

Number of Grid Openings: 20 Number of Grids Examined: 2 Average Area of Grid Opening (cm^2) .00006206

Total Area Examined (cm^2) .0012412 Detection Limit: 6,025 asbestos structures per m^3

Comments:

	Area Examined No.	Filtration Area No.	No./Volume No./L	No./CC
No. of Fibrous Structures (Total)	13	90,074	78.3	.08
No. of Chrysotile Structures	0	BDL**	--	--
No. of Amphibole Structures	11	76,217	66.3	.07
No. of Other* Structures	2	13,858	12.0	.01
No. of Fibers (Total)	12	83,145	72.3	.07
No. of Asbestos Fibers	10	69,288	60.2	.06
No. of Chrysotile Fibers	0	BDL	--	--
No. of Amphibole Fibers	10	69,288	60.2	.06
No. of Matrix/Debris (Asbestos)	0	BDL	--	--
No. of Cluster/Clumps (Asbestos)	1	6,929	6.0	.006
No. of Bundles (Asbestos)	0	BDL	--	--

* Category of "other" includes: Ambiguous, Non-Asbestos, and No E. D. Pattern.

** BDL = Below Detectable Limit.

Comments: Light loading

Figure 7. TEM asbestos analysis report.

QUALITY ASSURANCE

The objective of quality assurance activities is to provide quality data through the use of proper sampling procedures, careful handling of samples, the use of calibrated analytical equipment and standardized analytical protocols, and the checking of fiber analysis calculations. As standard procedure for tasks performed under this EPA contract, a comprehensive, written Quality Assurance Project Plan (QAPP) is prepared and submitted to EPA at least 30 days prior to the performance of any sampling, analyses, or data reductions. In this instance, however, the abatement schedules at the sites selected for monitoring and the dates of issuance of this task and work plan approval did not allow sufficient time for the preparation, review, and approval of a formal QAPP before the sampling had to begin. As a result, the requirement for submittal of a written QAPP was waived to take advantage of a unique opportunity to collect field data from these two readily accessible asbestos-abatement sites. Under these conditions, the following QA/QC criteria were incorporated into the scope of this project, and field and laboratory procedures were documented to ensure the integrity of the data generated.

Filter Preparation

All Millipore membrane filters used in the collection of asbestos air samples were preassembled by the manufacturer in three-piece plastic cassettes, and all were from the same production lot number. All the Nuclepore polycarbonate filters used for sampling also were from the same lot and were loaded into Millipore filter cassettes (on top of the 5.0-μm mixed-cellulose ester filter and cellulose backup pad) by laboratory personnel in a remote, clean area of the laboratory. The monitoring cassettes were reassembled, and a cellulose shrink band was placed around the base and middle stages of each cassette. The monitoring cassettes were labeled with a Field Sample ID Number prior to sampling.

Sampling

Constant-flow and/or rotameter-equipped personal sampling pumps were used to draw air through the filter/cassette assembly at a known flow rate of 2.5 liters/minute. Each pump was calibrated on site before sampling and checked after sampling by the use of a Magnehelic air flow gauge. Rotameters on the sampling pumps were checked periodically during the sampling period to ensure the constancy of the flow rate. No flow rate adjustments were required during the sampling periods.

The sampling strategy was to collect two or three pairs of samples in each completed work area (each pair consisting of one PCM and one TEM sample). The filter cassettes were positioned about 5 feet (1.5 m) above the floor and were supported by taping the end of the hose to the wall or clipping it to a tripod. The sampling locations were determined arbitrarily rather than randomly. The sampling strategy was to have at least one sampling location in the center of the area and one or two near the perimeter.

Field record books were maintained by the onsite field technicians or supervisor at each sampling site. Air Monitoring Data Sheets (Figure 8) were used to record the following information for each series of air tests:

- Sampling site
- Date and time
- Location of sampling equipment
- Sample number
- Sample type
- Sampling method
- Sampling parameters (flow rates, start time, stop time, duration)
- Field technicians' observations

Upon completion of sampling, each filter cassette was turned upright (i.e., the filter plane was parallel to the floor), the pump was turned off, the face cap was positioned tightly on the filter cassette, and the cassette was disconnected from the sampling hose. The filter cassettes were hand-carried to the appropriate laboratory for analysis or to an intermediate site for carbon-coating, and then carried or shipped via courier to the analytical lab.

The Millipore filters for PCM analysis were checked into the laboratory, where each sample was assigned an alphanumeric identity code. The Nuclepore samples were submitted to the EPA Project Officer (or his representative) and later sent to the TEM laboratory.

Chain of Custody

A chain-of-custody form (Figure 9) was filled out in ink for each set of samples collected in the field. Each form was initiated by the onsite field technician who collected the samples. The next person having custody of the samples noted receipt of the samples and completed the appropriate section of the form. As standard procedure, samples arriving at the PEI laboratory are checked in by the laboratory sample custodian, who examines the shipping container and each filter cassette for any evidence of damage or tampering, notes any damage or indication of tampering on the enclosed chain-of-custody form, and then signs the form. Once samples are received by the laboratory, a similar intralaboratory chain-of-custody procedure is maintained so that the location of the samples and the person having custody are always known.

Sample Analysis

The PCM samples were analyzed in accordance with NIOSH Method No. P&CAM 239. All microscopists performing PCM analyses have successfully completed NIOSH Course No. 582, Sampling and Evaluating Airborne Asbestos Dust, and have participated in the NIOSH Proficiency Analytical Testing (PAT) Program. As part of the American Industrial Hygiene Association's laboratory accreditation program, the laboratory that conducted the PCM analyses participates in the NIOSH PAT Program. This program currently includes biomonthly analyses for asbestos and other occupational contaminants. Known reference standards (i.e, PAT asbestos samples) are routinely analyzed to ensure the accuracy of

Sheet No. _____

AIR SAMPLING DATA SHEET

DATE_____

Facility _____

Address _____

City _____ State _____ Zip _____

Sample No.	Location	Equipment Type	Equipment ID	Flow rate, ___/min	Time On	Time Off	Time Net	Volume, liters	Units, _____	Conc., _____

Figure 8. Air sampling data sheet.

SAMPLE SHIPPING/RECEIVING RECORD

1. NAME OF ESTABLISHMENT _____ PN _____

2. SENDER	3. CARRIER	4. RECEIVER
Signature _____	Company _____	Courier from Depot
Date _____	Signature _____	Signature _____
Sent from _____	Date _____	Date _____
_____	B/L No. _____	LAB CUSTODIAN
_____		Signature _____
		Date _____
		Condition upon _____
		Receipt _____
		_____

5. SHIPMENT DESCRIPTION

	Seal No.	Seal intact?	Seal No.	Seal intact?
Number of packages _____				
Sealed (yes or no) _____				
Types of containers _____				
_____				
Condition prior to shipment _____				
_____				

6. CONTENTS

Sample I.D. number	Type of sample	Sealed (yes or no)	Seal No. if any	Condition (damaged, loss of liquid, etc.)

Figure 9. Chain-of-custody form.
(Sample shipping/receiving record).

the PCM results. The results of the analysis of PAT asbestos reference samples have consistently fallen within the acceptable limits of variation for the method. A record of the precision of the PCM analysis is generally kept by calculating the coefficient of variation of the results of replicate analyses. One sample blank is analyzed for every 10 samples to check the quality of the filter media and sample preparation procedure.

All data generated by the laboratory and by the field technician during this task were checked for technical accuracy by the laboratory supervisor or the certified industrial hygienist. This involved verifying that the mathematical computations were correct and that the appropriate formulae were used. The laboratory supervisor reported the analytical data in writing to the project manager.

The standard QA/QC procedures that apply to the Nuclepore samples submitted for TEM analysis are as defined by IITRI's QA/QC plan for TEM analysis.

SECTION 6

RESULTS

AIR MONITORING RESULTS

Table 1 presents a detailed listing of the results of PCM and TEM analysis of samples collected during aggressive and nonaggressive sampling conditions after abatement. With one exception, all concentrations of asbestos fibers and total structures under aggressive sampling conditions were higher than the corresponding measurements made under nonaggressive sampling conditions. Only one PCM sample collected under nonaggressive sampling conditions (Sample No. COR-02) had a fiber concentration that was higher than the corresponding PCM aggressive sample (Sample No. COR-27). The difference between these two samples (0.007 versus 0.002 fibers/m^3) is considered negligible because both sample results were below the limit of reliable quantitation for the analytical method. Figure 10 presents a summarized version of the air monitoring data on a floor plan of the building in which the samples were collected.

Comparisons of the results of PCM and TEM analyses of samples collected during nonaggressive and aggressive sampling conditions are presented graphically in Figure 11, which is based on the data results presented in Table 1.

STATISTICAL COMPARISONS

Statistical Method of Analysis

The Mann-Whitney test was used to determine whether the observed differences in analytical methods, sampled areas, and sampling conditions were statistically significant.[9] Use of the Mann-Whitney test required no a priori assumption regarding the nature of the underlying probability distribution function of measurements of asbestos fiber concentrations. A detailed discussion of the Mann-Whitney test and an example of its application are presented in Appendix C.

Analytical Methods

Tables 2 and 3 present comparisons between the geometric averages of fiber concentrations determined by PCM and TEM analyses under nonaggressive and aggressive sampling conditions. Table 4 presents a summary of these results. Based on the application of the Mann-Whitney test and the assumption that the "fiber"/volume concentrations are comparable, the difference between PCM and TEM results is statistically significant (i.e., $p < 0.03$) for samples collected outdoors and indoors in abated areas under nonaggressive conditions.

TABLE 1. RESULTS OF PCM & TEM ANALYSES

Sampling location	Nonaggressive					Aggressive				
	PCM		TEM			PCM		TEM		
	Sample number	10^3 fibers/m³	Sample number	10^6 asbestos[a] fibers/m³	10^6 asbestos[b] structures/m³	Sample number	10^3 fibers/m³	Sample number	10^6 asbestos[a] fibers/m³	10^6 asbestos[b] structures/m³
Abated areas										
Room 155	CBR-02	0.007[c]	CBR-A-02	0.006	0.006	CBR-27	0.005[c]	CBR-A-27	0.017	0.022
	CBR-03	0.002[c]	CBR-A-03	0.016	0.016	CBR-25	0.01[c]	CBR-A-25	0.041	0.041
	CBR-01	<0.002[d]	CBR-A-01	0.010	0.010	CBR-26	0.007[c]	CBR-A-26	0.024	0.024
Room 157	CBR-04	0.008[c]	CBR-A-04	0.018	0.028	CBR-28	0.032	CBR-A-28	0.041	0.041
	CBR-05	0.009[c]	CBR-A-05	0.017	0.017	CBR-29	0.040	CBR-A-29	0.104	0.104
	CBR-06	0.01[c]	CBR-A-06	0.012	0.012	CBR-30	0.035	CBR-A-30	0.059	0.065
Room 159	CBR-08	<0.002[d]	CBR-A-08	<0.005[e]	<0.005[e]	CBR-31	0.045	CBR-A-31	0.060	0.066
	CBR-07	<0.002[d]	CBR-A-07	0.006	0.006	CBR-46	0.045	CBR-A-45	0.025	0.025
	CBR-09	0.004[c]	CBR-A-09	0.011	0.011	CBR-34	0.057	CBR-A-34	0.022	0.028
Nonasbestos areas										
Room 173	CBR-13	<0.002[d]	CBR-A-13	0.011	0.011	CBR-35	0.004[c]	CBR-A-35	0.006	0.006
	CBR-14	0.002	CBR-A-14	†	†	CBR-36	0.003[c]	CBR-A-36	†	†
Room 152	CBR-10	<0.002[d]	CBR-A-10	0.012	0.012	CBR-37	0.17	CBR-A-37	0.135	0.149
	CBR-16	0.002	CBR-A-16	†	†	CBR-38	0.18	CBR-A-38	†	†
Room 205	CBR-19	<0.002[d]	CBR-A-19	0.024	0.024	CBR-40	0.01[c]	CBR-A-40	0.032	0.032
	CBR-11	<0.002[d]	CBR-A-11	†	†	CBR-39	0.006[c]	CBR-A-39	†	†
	CBR-17	0.005[c]	CBR-A-17	†	†	CBR-41	0.01[c]	CBR-A-41	†	†
Outdoors (ambient)										
Ground	CBR-21	<0.002[d]	CBR-A-21	<0.006[e]	<0.006[e]					
	CBR-44	<0.002[d]	CBR-A-44	†	†					
Roof	CBR-43	<0.002[d]	CBR-A-43	0.011	0.011					
	CBR-18	<0.002[d]	CBR-A-18	†	†					
	CBR-42	<0.002[d]	CBR-A-33	†	†					
Blanks	CBR-45	0/100[g]	CBR-A-60	0/20[h]	0/20[h]					
	CBR-47	0/100[g]	CBR-A-61	1/20[i]	1/20[i]					
	CBR-48	0/100[g]								
	CBR-49	0/100[g]								

[a] Fiber concentration based on the total number of asbestos fibers counted.

[b] Concentration based upon the total number of chrysotile and amphibole structures counted. These asbestos structures include asbestos fibers, asbestos matrices/debris, asbestos clusters/clumps, and asbestos bundles.

[c] Less than 10 fibers in 100 fields were counted. Fiber concentration based on the actual number of fibers counted in 100 fields. Fiber concentration is below the reliable limit of quantitation (i.e., 10 fibers in 100 fields).[f] Sample calculation:

$$\text{Lower limit of reliable quantitation} = \frac{10 \text{ fibers}/100 \text{ fields}}{\text{volume of air sampled (m}^3\text{)}} \times \frac{\text{effective collecting area of the filter (mm}^2\text{)}}{\text{microscopic field area (mm}^2\text{)/field}}$$

$$= \frac{10 \text{ fibers}/100 \text{ fields}}{1.200 \text{ m}^3} \times \frac{855 \text{ mm}^2/\text{filter}}{0.003136 \text{ mm}^2/\text{field}} = 22{,}720 \text{ fibers/m}^3$$

[d] No fibers were detected in 100 fields. Below the detection limit (e.g., counting 0.5 fiber in 100 fields). Sample calculation:

$$\text{Detection limit} = \frac{0.5 \text{ fiber}/100 \text{ fields}}{\text{volume of air sampled (m}^3\text{)}} \times \frac{\text{(Effective collecting area of the filter (mm}^2\text{)}}{\text{Microscopic field area (mm}^2\text{)/field}}$$

$$= \frac{0.5 \text{ fiber}/100 \text{ fields}}{1.200 \text{ m}^3} \times \frac{855 \text{ mm}^2/\text{filter}}{0.003136 \text{ mm}^2/\text{field}} = 1136 \text{ fibers/m}^3$$

[e] Below detection limit (no fibers or structures counted in 20 grid openings). Sample calculation:

$$\text{Detection limit} = \frac{1 \text{ fiber}}{\text{No. of grid openings scanned}} \times \frac{\text{Area of filter (cm}^2\text{)}}{\text{area of grid opening (cm}^2\text{)}} \times \frac{1}{\text{volume of air (m}^3\text{)}}$$

$$= \frac{1 \text{ fiber}}{20} \times \frac{8.6 \text{ cm}^2}{0.63 \times 10^{-4} \text{ cm}^2} \times \frac{1}{1.200 \text{ m}^3} = 5680 \text{ fibers/m}^3$$

[f] Sample collected but not analyzed.

[g] No fibers were detected in 100 fields.

[h] No asbestos fibers or structures were detected in 20 grid openings.

[i] One asbestos fiber or structure was detected in 20 grid openings.

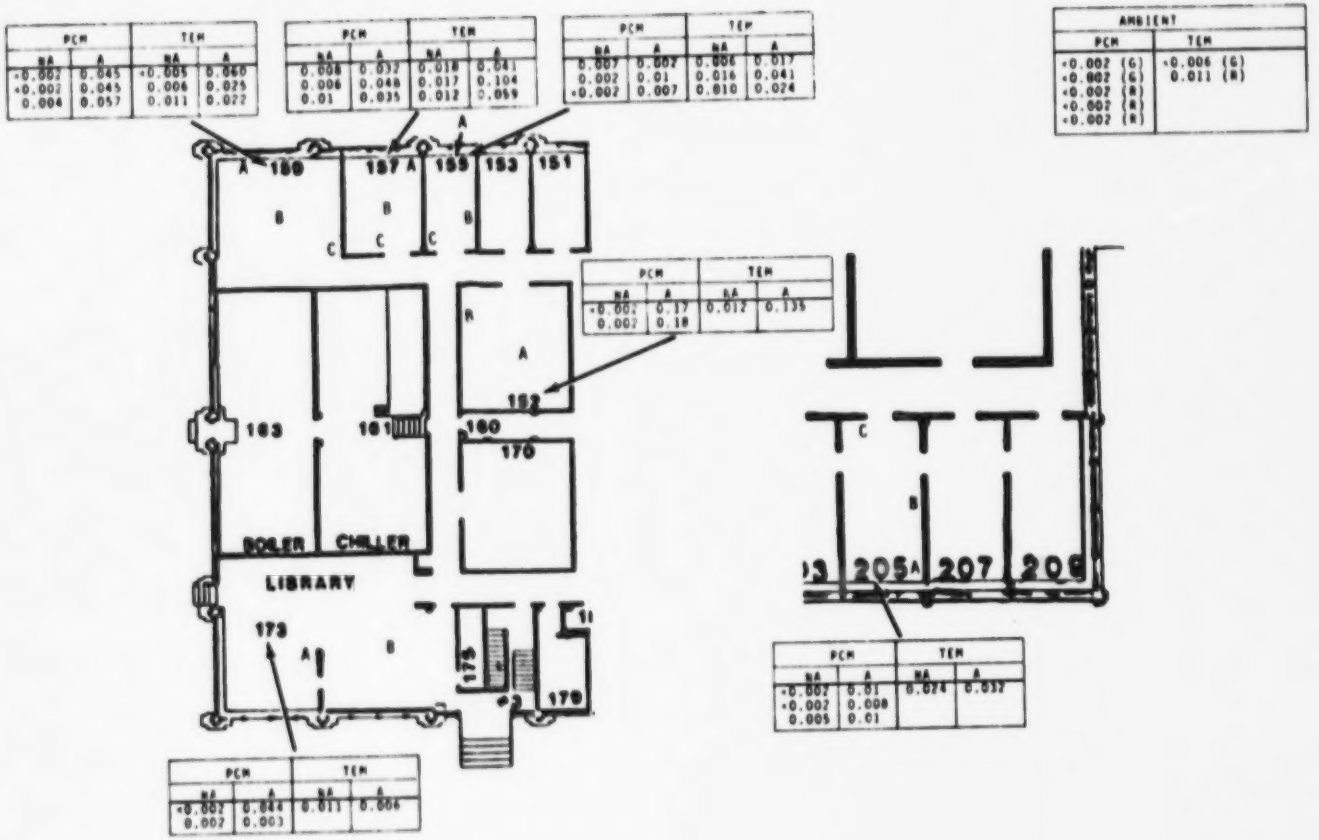

Figure 10. Results of phase-contrast and transmission-electron microscopy analyses from nonaggressive (NA) and aggressive (A) sampling conditions.

Note: All concentrations are fibers/cm³. Data are listed by location A through C (top to bottom).

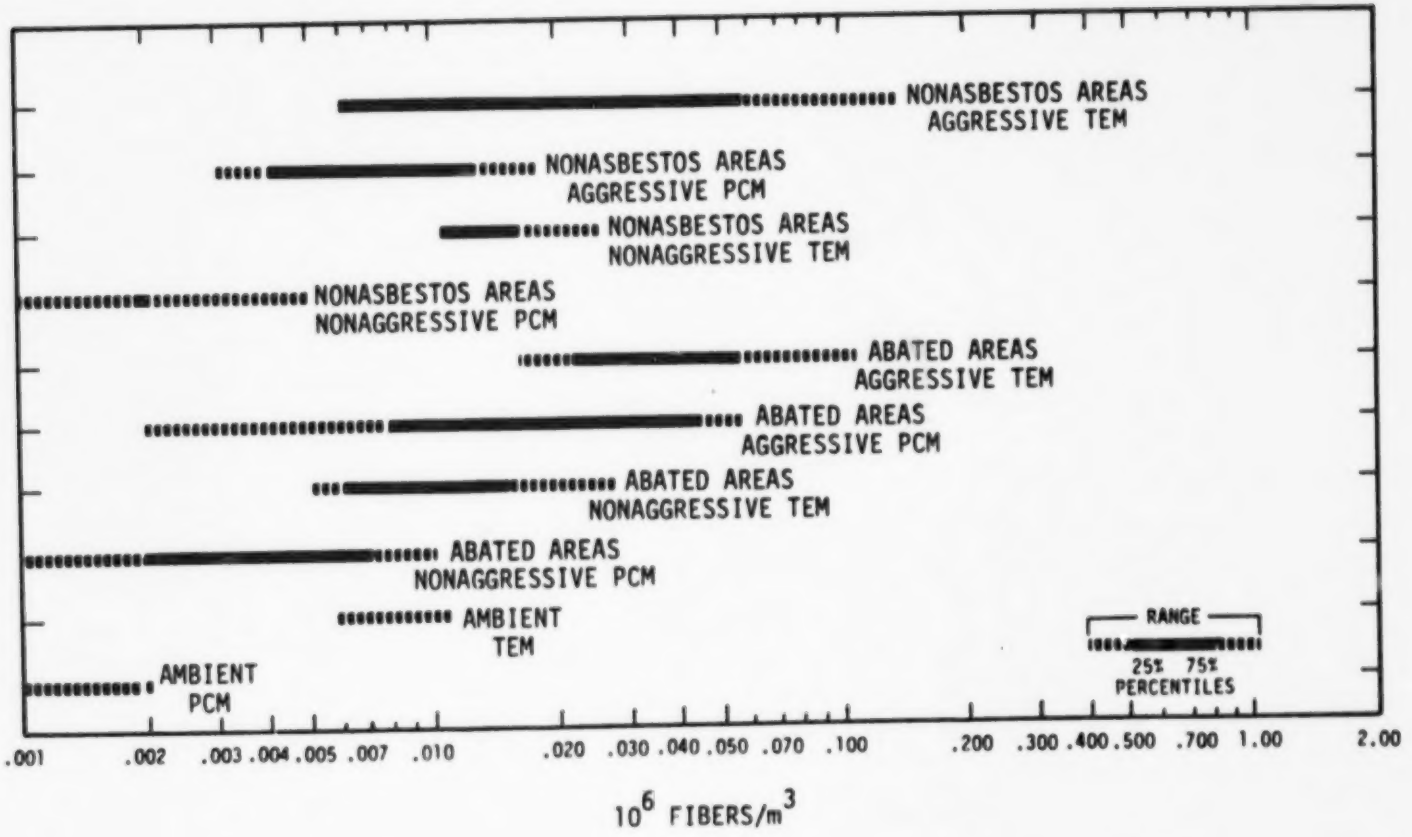

Figure 11. Comparison of airborne fiber concentration.

TABLE 2. COMPARISON OF NONAGGRESSIVE AND AGGRESSIVE SAMPLING RESULTS FOR POSTABATEMENT TESTING

Samples included in comparison	Nonaggressive						Aggressive						PCM Aggressive/ Nonaggressive fibers,[a] $10^6/m^3$	TEM Aggressive/ nonaggressive	
	PCM		TEM				PCM		TEM						
	No. of samples	Fibers,[a] $10^6/m^3$	No. of samples	Asbestos fibers,[a] $10^6/m^3$	Asbestos structures,[a] $10^6/m^3$	TEM/PCM fibers	No. of samples	Fibers,[a] $10^6/m^3$	No. of samples	Asbestos fibers,[a] $10^6/m^3$	Asbestos structures,[a] $10^6/m^3$	TEM/PCM fibers		asbestos fibers	asbestos structures
Abated areas	9	0.003	9	0.010	0.010	3.3	9	0.021	9	0.037	0.040	1.8	7.0	3.7	4.0
Nonasbestos areas	7	<0.002	3	0.015	0.015	7.5	7	0.016	3	0.030	0.031	1.9	8.0	2.0	2.1
Outdoors	5	<0.002	2	0.006	0.006	3.0	0	-	0	-	-	-	-	-	-

[a]All concentrations are geometric means.

TABLE 3. COMPARISON OF SAMPLING RESULTS BY SAMPLE LOCATION[a]

Samples included in comparison	Sample location comparisons		
	Indoor abated/ indoor nonasbestos	Indoor abated/ outdoors	Indoor nonasbestos/ outdoors
PCM-Nonaggressive	3.0	3.0	1.0
PCM-Aggressive	1.3	3.5	16.0
TEM-Nonaggressive	0.7 (0.7)[b]	1.7 (1.7)[b]	2.5 (2.5)[b]
TEM-Aggressive	1.2 (1.3)[b]	6.2 (6.7)[b]	5.0 (5.2)[b]

[a]All quantities are ratios of the geometric mean fiber concentrations. For PCM samples, fiber concentrations include all fibers greater than 5 μm in length; for TEM samples, fiber concentrations include all asbestos fibers.

[b]Ratio of geometric mean concentrations of asbestos structures.

TABLE 4. SUMMARY COMPARISON OF PCM AND TEM ANALYSES OF AIR SAMPLES COLLECTED DURING NONAGGRESSIVE AND AGGRESSIVE CONDITIONS[a]

Analytical technique	Outdoor (ambient)	Nonasbestos areas		Abated areas	
		Nonaggressive	Aggressive	Nonaggressive	Aggressive
Phase contrast microscopy (PCM), fibers (> 5 μm)/m³	BDL[b]	BDL	BLRQ[c] (16,000)	BLRQ (3,000)	BLRQ (21,000)
Transmission electron microscopy (TEM)					
asbestos fibers/m³	6,000[d]	15,000	30,000	10,000	37,000
asbestos structures/m³	6,000[d]	15,000	31,000	10,000	40,000

[a] All values are geometric means.

[b] BDL = Below detection limit (≈1,136 fibers/m³).

[c] BLRQ = Below limit of reliable quantitation (≈22,720 fibers/m³).

[d] Geometric mean based on two sample values. One sample value was below the detection limit for TEM analysis (≈5,688 asbestos fibers or structures/m³).

The difference between PCM and TEM results from indoor sampling of nonasbestos areas was also statistically significant ($p < 0.10$) under nonaggressive sampling conditions; however, this conclusion was based on a small sample size ($n = 3$). The ratios of TEM/PCM concentrations for nonaggressive sampling were 3.0 for ambient samples, 3.3 for indoor abated-area samples, and 7.5 for indoor nonasbestos-area samples. The difference between PCM and TEM results is not statistically significant (i.e., $p > 0.05$) for indoor samples from both abated and nonasbestos areas under aggressive sampling conditions. For aggressive sampling in abated areas, the ratio of TEM/PCM was 1.8. For aggressive sampling in nonasbestos areas, the ratio of TEM/PCM was 1.9.

Sampling Conditions

Table 2 also provides a comparison of nonaggressive and aggressive sampling conditions for PCM and TEM analyses in both abated and nonasbestos areas. The difference between the geometric average fiber concentrations under nonaggressive and aggressive sampling conditions was statistically significant (i.e., $p < 0.002$) for both PCM and TEM in abated areas. For PCM analyses, the ratio of aggressive to nonaggressive fiber concentrations was 7.0; for TEM analyses, the ratio was 3.7. For sampling conducted in nonasbestos areas, the difference between the geometric average fiber concentrations under nonaggressive and aggressive sampling conditions was statistically significant for PCM analyses (i.e., $p < 0.002$), but not statistically significant for TEM analyses (i.e., $p > 0.4$). For nonasbestos areas, the ratio of aggressive to nonaggressive fiber concentrations for PCM analyses was 8.0; for TEM analyses, the ratio was 2.0.

Appendix D presents a comparison of TEM results from two air samples taken from the same location during nonaggressive and aggressive conditions. These figures exemplify the findings of this study, i.e., that the concentrations of asbestos fibers and structures measured under aggressive sampling conditions are higher than those measured under nonaggressive conditions.

Comparison of Indoor Abated Samples and Ambient Samples

Also included in Tables 2, 3, and 4 are the PCM and TEM analyses of samples collected in the ambient atmosphere for comparison with samples collected in indoor abated areas. For samples analyzed by PCM, the geometric mean fiber concentration was 0.003×10^6 fibers/m^3 for indoor samples (nonaggressive) in abated areas compared with less than 0.002×10^6 fibers/m^3 for ambient samples. The PCM method, however, is not sufficiently sensitive for reliable detection of these ambient and indoor (nonaggressive) concentrations. All ambient concentrations were below the detection limit of the PCM method and concentrations in indoor abated areas were below the lower limit of reliable quantitation by this method. Consequently, the observed differences between the two sample groups are probably not meaningful.

For the TEM samples collected inside abated areas (under nonaggressive conditions), the geometric mean asbestos fiber concentration was 0.010×10^6 fibers/m^3 compared with 0.006×10^6 fibers/m^3 for ambient samples--a ratio of

1.7. The observed difference between these indoor, nonaggressive, TEM asbestos fiber concentrations and the ambient TEM asbestos fiber concentrations was not statistically significant ($p > 0.15$). The difference between asbestos fiber concentrations under aggressive sampling conditions in indoor abated areas and ambient asbestos concentrations was statistically significant ($p < 0.02$). The ratio of asbestos fiber concentrations by TEM under aggressive sampling in indoor abated areas to ambient TEM fiber concentrations was 6.2.

Comparison of Indoor Nonasbestos Samples and Ambient Samples

For samples analyzed by PCM, the geometric mean fiber concentration for indoor samples collected nonaggressively in nonasbestos areas was below the detection limit of the analytical method, as were the ambient PCM samples. Consequently, no meaningful comparisons can be made. For PCM samples collected aggressively, the geometric mean fiber concentration was 0.016×10^6 fibers/m^3 compared with less than 0.002×10^6 fibers/m^3 for ambient samples, a ratio of 16.0 (if a concentration of 0.001×10^6 fibers/m^3 for ambient samples is assumed). The observed difference between aggressive (nonasbestos area) PCM fiber concentrations and the ambient PCM fiber concentrations was statistically significant ($p < 0.01$). One nonasbestos area (Room 152, Instrumentation Laboratory) was extremely dusty. The aggressive sampling procedure entrained large quantities of "house dust" that had accumulated on shelf and cabinet tops over many years. This accounted for the relatively high PCM fiber counts.

For TEM samples collected inside nonasbestos areas (under nonaggressive conditions), the geometric mean asbestos fiber concentration was 0.015×10^6 fibers/m^3 compared with 0.006×10^6 fibers/m^3 for TEM ambient samples, a ratio of 2.5. The observed difference between these nonaggressive, indoor (nonasbestos area) TEM asbestos fiber concentrations and the ambient TEM asbestos fiber concentrations was not statistically significant (i.e., $p > 0.10$). The difference between TEM asbestos fiber concentrations under aggressive sampling conditions in indoor nonasbestos areas and ambient TEM asbestos concentrations was not statistically significant (i.e., $p > 0.10$). The ratio of asbestos fiber concentrations determined by TEM under aggressive sampling conditions inside nonasbestos areas to ambient TEM fiber concentrations was 5.0. Because the comparisons between fiber concentrations for TEM samples in nonasbestos areas (aggressive or nonaggressive) and ambient samples are based upon very small sample sizes ($n = 3$ and $n = 2$, respectively), the observed differences in fiber concentrations between these two sampled locations cannot be found statistically significant at a probability level of < 0.05.

Comparison of Samples From Indoor Abated and Indoor Nonasbestos Areas

For all PCM samples (aggressive and nonaggressive), the observed difference between fiber concentations in indoor abated areas and indoor nonasbestos areas was not statistically significant ($p > 0.08$ for nonaggressive conditions; $p > 0.05$ for aggressive conditions). For PCM samples collected under nonaggressive conditions, the ratio of fiber concentrations in abated

areas to nonasbestos areas was 3.0 (a concentration of 0.001 x 10^6 fibers/m^3 for indoor nonasbestos samples was assumed since the mean geometric concentration in indoor abated areas was less than 0.002 x 10^6 fibers/m^3). For PCM samples collected under aggressive conditions, the ratio of fiber concentrations in abated areas to nonasbestos areas was 1.3.

For all samples analyzed by TEM, the difference between abated and nonasbestos areas was also not statistically significant ($p > 0.10$ for nonaggressive conditions; $p > 0.10$ for nonagressive conditions). For TEM samples collected under nonaggressive conditions, the ratio of asbestos fiber concentrations in abated areas to nonasbestos areas was 0.7. For TEM samples collected under aggressive conditions, this ratio was 1.2 (1.3 for asbestos structures).

Expanded TEM Data From Nonaggressive and Aggressive Sampling Conditions

Table 5 presents additional data from the TEM asbestos analysis reports, including the types of asbestos fibers observed, the number of other fibrous structures, and the number of nonfibrous asbestos particles (i.e, matrix/debris, cluster/clumps, and bundles). The relationship and significance of these other parameters, as influenced by different sampling conditions and monitoring locations, were not evaluated as part of this study.

TABLE 5. EXPANDED TEM DATA FROM NONAGGRESSIVE AND AGGRESSIVE SAMPLING CONDITIONS

Sampling location	Sample number	Asbestos, 10^6 fibers/m^3	10^6 asbestos structures/m^3	Nonaggressive: Concentration, 10^6 units/m^3 — Tot asb fib	Chry fib	Amp fib	Tot fib	Tot fib structures	Chry structures	Amp structures	Other structures	Asb mat/deb	Asb clus/clump	Asb bundles
Abated areas														
Room 155														
A	2	0.006	0.006	0.006	ND[a]	0.006	0.01	0.01	ND	0.006	0.006	ND	ND	ND
B	3	0.02	0.015	0.02	0.005	0.01	0.02	0.03	0.005	0.01	0.01	ND	ND	0.005
C	1	0.01	0.01	0.01	ND	0.01	0.03	0.03	ND	0.01	0.02	ND	0.005	ND
Room 157														
A	4	0.03	0.03	0.03	0.01	0.02	0.03	0.03	0.01	0.02	0.006	ND	ND	ND
B	5	0.02	0.016	0.02	0.01	0.006	0.02	0.02	0.01	0.006	0.006	ND	ND	ND
C	6	0.01	0.01	0.01	ND	0.01	0.01	0.01	ND	0.01	ND	ND	ND	ND
Room 159														
A	8	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
B	7	0.006	0.006	0.006	0.006	ND	0.006	0.006	0.006	ND	ND	ND	ND	ND
C	9	0.01	0.01	0.01	ND	0.01	0.03	0.03	ND	0.01	0.02	ND	ND	ND
Nonasbestos areas														
Room 173														
A	13	0.01	0.01	0.01	ND	0.01	0.03	0.03	ND	0.01	0.02	ND	ND	ND
B	14	Sample not analyzed												
Room 152														
A	10	0.01	0.01	0.01	ND	0.01	0.02	0.02	ND	0.01	0.006	ND	ND	ND
B	16	Sample not analyzed												
Room 205														
A	11	Sample not analyzed												
B	19	0.02	0.02	0.02	ND	0.02	0.03	0.03	ND	0.02	0.006	ND	ND	ND
C	17	Sample not analyzed												
Outdoors (ambient)														
Ground	21	ND	ND	ND	ND	ND	ND	0.03	ND	ND	0.03	ND	ND	ND
	22	Sample not analyzed												
	44	Sample not analyzed												
	45	Sample not analyzed												
Roof	18	Sample not analyzed												
	20	Sample not analyzed												
	42	Sample not analyzed												
	33	Sample not analyzed												
	43	0.01	0.01	0.01	ND	0.01	0.01	0.01	ND	0.01	ND	ND		
Sample blanks	60	ND	ND	ND	ND	ND	ND	1[b]	ND	ND	1[b]	ND	ND	ND
	61	ND	ND	ND	ND	ND	ND	1[b]	ND	ND	1[b]	ND	ND	ND
													ND	ND

[a] ND = None detected in the grid openings examined.

[b] One nonasbestos fibrous structure was detected.

(continued)

TABLE 5 (continued)

Sampling location	Sample number	Asbestos, 10^6 fibers/m^3	10^6 asbestos structures/m^3	Aggressive: Concentration, 10^6 units/m^3										
				Tot asb fib	Chry fib	Amp fib	Tot fib	Tot fib structures	Chry structures	Amp structures	Other structures	Asb mat/deb	Asb clus/clump	Asb bundles
Abated areas														
Room 155														
A	27	0.02	0.026	0.02	0.006	0.01	0.04	0.05	0.006	0.02	0.03	ND	ND	0.006
B	25	0.04	0.04	0.04	0.02	0.02	0.05	0.06	0.02	0.02	0.02	0.006	ND	ND
C	26	0.02	0.02	0.02	ND	0.02	0.03	0.03	ND	0.02	0.006	ND	ND	ND
Room 157														
A	28	0.04	0.04	0.04	0.01	0.03	0.05	0.06	0.01	0.03	0.01	ND	ND	ND
B	29	0.10	0.106	0.10	0.006	0.10	0.16	0.16	0.006	0.10	0.05	ND	ND	ND
C	30	0.06	0.06	0.06	ND	0.06	0.08	0.08	ND	0.06	0.02	0.006	ND	ND
Room 159														
A	31	0.06	0.07	0.06	ND	0.06	0.07	0.08	ND	0.07	0.01	ND	0.006	ND
B	46	0.03	0.03	0.03	ND	0.03	0.06	0.06	ND	0.03	0.03	ND	ND	ND
C	34	0.02	0.03	0.02	ND	0.02	0.05	0.06	ND	0.03	0.03	0.006	ND	ND
Nonasbestos areas														
Room 173														
A	35	0.006	0.006	0.006	ND	0.006	0.02	0.02	ND	0.006	0.01	ND	ND	ND
B	36	Sample not analyzed												
Room 152														
A	37	0.01	0.01	0.01	ND	0.01	0.02	0.02	ND	0.01	0.01	0.01	ND	ND
B	38	Sample not analyzed												
Room 205														
A	39	Sample not analyzed												
B	40	0.03	0.03	0.03	ND	0.03	0.06	0.06	ND	0.03	0.03	ND	ND	ND
C	41	Sample not analyzed												
Outdoors (ambient)														
Ground														
Roof														
Sample blanks														

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APPENDIX A

METHODOLOGY FOR THE MEASUREMENT OF AIRBORNE ASBESTOS BY TRANSMISSION ELECTRON MICROSCOPY - LEVEL II ANALYSIS PROTOCOL*

LEVEL II ANALYSIS*

SUMMARY OF PROTOCOL

Level II analysis is a regulatory technique consisting of Level I analysis plus chemical elemental analysis. Morphology, size, SAED pattern, and chemical analysis are obtained sequentially. By a process of elimination, mineral fibers are identified as chrysotile, amphibole, ambiguous, or "no-identity" by morphology and SAED pattern. X-ray elemental analysis is used to categorize the amphibole fibers, identify the ambiguous fibers, and confirm or validate chrysotile fibers.

Level II analysis is summarized as follows:

(1) A known volume of air is passed through a polycarbonate membrane filter (pore diameter, 0.4 μm; filter diameter, 37 or 47 mm) to obtain approximately 5 to 10 μg of particulates per cm^2 of filter surface.

(2) The particulate-laden filter is transported in its own filter holder.

(3) The filter is carbon-coated in the holder.

(4) The particulates are transferred to an EM grid using a refined Jaffe wick washer.

(5) The EM grid, containing the particulates, is gold-coated lightly.

(6) The EM grid is examined under low magnification (250X to 1000X) followed by high-magnification (16,000X on the fluorescent screen) search and analysis.

(7) A known area (measured grid opening) is scanned, and the asbestos structures (fibers, bundles, clusters, and matrices) are counted, sized, and identified as to asbestos type (chrysotile, amphibole, ambiguous, or no identity) by morphology and by observing the SAED pattern; and finally by elemental analysis using EDS.

(8) The observations are recorded--a minimum of 100 fibrous structures or 10 grid openings, whichever is first.

(9) The data are reduced and the results reported.

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* Reprinted from Yamate, G., S. C. Agarwal, and R. D. Gibbons. 1984. Methodology for the Measurement of Airborne Asbestos by Electron Microscopy. Draft Report. Office of Research and Development, U.S. Environmental Protection Agency, Washington, D.C. Contract No. 68-02-3266.

EQUIPMENT, FACILITIES, AND SUPPLIES

The following items are required for Level I analysis:

(1) A modern 100-kV TEM equipped with an EDS. A scanning accessory as found in a STEM will increase the versatility and analytical capability for very small fibers and for fibers adjacent to other particulate matter. The microscope should be equipped with the fluorescent viewing screen inscribed with graduation of known radii to estimate the length and width of fibrous particulates.

(2) A vacuum evaporator with a turntable for rotating specimens during coating, for such uses as carbon-coating polycarbonate filters, gold-coating EM grids, and preparing carbon-coated EM grids.

(3) An EM preparation room adjacent to the room housing the EM. This room should either be a clean-room facility, or contain a laminar-flow class-100 clean bench to minimize contamination duing EM grid preparation. Filter handling and transfer to EM grids should be performed in a clean atmosphere. Laboratory blanks should be prepared and analyzed weekly to ensure quality of work.

(4) Several refined Jaffe wick washers for dissolving membrane filters.

(5) Miscellaneous EM supplies and chemicals, including carbon-coated 200-mesh copper grids, grid boxes, and chloroform.

(6) Sample collection equipment, including 37-mm-diameter or 47-mm-diameter filter holders, 0.4-μm (pore size) polycarbonate filters, 5.0-μm (pore size) cellulose ester membrane filters for back-up, a sampling pump with ancillary equipment, a tripod, critical orifices or flow meters, and a rain/wind shield.

DESCRIPTION OF METHODOLOGY

1. Type of Samples--Source

This protocol is an expansion of the method originally developed for the EPA for measuring airborne asbestos (Samudra et al., 1977; Samudra et al., 1978). A broad interpretation of airborne has been to apply the term to samples obtained from ambient air (the original purpose), aerosolized source materials (such as the asbestos workplace environment, and fugitive dust emissions), bulk-air material (such as total suspended particulate (TSP) samples, dust, and powders) and any other type of sample obtained by nonrestrictive use of (1) collection of a volume of air, (2) separation from the air, and (3) concentration of the particulates onto a substrate. The airborne protocol has also been applied to samples collected in the regulatory areas of the EPA, as compared with, for example, the workplace environment (National

Institute of Occupational Safety and Health), mining activities (U.S. Bureau of Mines), and shipboard atmosphere (Federal Maritime Administration).

The present methodology has been optimized for application specifically to samples collected from a volume of air in which the asbestos concentration is considered a minor component of the total particulate loading (other analytical methods are available for samples known to contain high concentrations of asbestos); and in which the particles are less than 15 μm in diameter, since particles greater than 15 μm either are not inhaled or are deposited in the upper respiratory tract and expelled, and preferably less than 10 μm in diameter as recommended by the Clean Air Scientific Advisory Committee (Hileman, 1981), since particles up to 10 μm can be absorbed by the alveolar region of the lung. These concentration and size restrictions will preclude many air samples collected in an asbestos-processing environment and in bulk-air material from the complete methodology. However, such samples can still be examined with the TEM, within the limitations of the instrument by changes in preparation techniques--provided the effects on the final results, such as fractionation of size and representativeness of the sample, are carefully considered.

2. Sample Collection and Transport

Sample Collection—

Sampling procedures vary depending on the nature of the sample, purpose of collection, analytical method to be used, sample substrate, and time and cost of sample collection relative to the total analytical effort. Nevertheless, the primary objective of sample collection always is to obtain a representative, unbiased sample.

Impingers, impaction devices, electrostatic precipitators, and thermal precipitators have been used in sample collection, but each has limitations. Presently, the preferred substrates are membrane filters, which are manufactured from different polymeric materials, including polycarbonate, mixed esters of cellulose, polystyrene, cellulose acetate, and cellulose nitrate. Polycarbonate membrane filters differ from the others in being thin, strong, and smooth-surfaced, and in having sieve-like construction (circular pores from top surface to the bottom). The other membrane filters are thicker, have irregular-surfaces, and have depth-filter construction (tortuous paths from top surface to bottom).

Consequently, polycarbonate filters have been selected for airborne asbestos analysis. The collection of small-sized particles (prefer less than 10 μm in diameter), the light loading of particulates, the uniform distribution of particulates attainable using a depth-type backing filter, the smooth surface and circular holes (which aid in determining size and instrument tilt axis), and the relative ease in grid transfer (thin and strong) minimize disadvantages of lack of retention and/or movement of large particles during handling. Other membrane materials, such as the cellulose ester type, are recommended for phase contrast and PLM, heavy particle loadings, and physical retention of large particles.

In microscopical analysis, uniformity of particulate distribution and loading is critical to success. Air samples are taken on 37-mm-diameter or 47-mm-diameter, 0.4-μm (pore size) polycarbonate membrane filters using the shiny, smooth side as the particle-capture surface. Cellulose ester-type membrane filters (pore size, 5.0 μm) are used to support the polycarbonate filter on the support pad (37-mm-diameter personal sampler) or on the support plate (47-mm-diameter holder).

Field monitoring cassettes (37-mm-diameter) of three-piece construction are available from several manufacturers. As with the 47-mm-diameter filters, loading the cassettes with the support pad, back-up filter, and 0.4 μm (pore size) polycarbonate filter should be carefully performed on a class-100 clean bench. Since the filters are held in place by pressure fit rather than by screw tightening, air must not enter from the sides of the unit; a plastic band or tape (which can double as a label) should be used as a final seal.

Collecting airborne samples with proper loading requires experience. Each of the following techniques is useful in collecting airborne samples for direct microscopy, preserving representative sizes, without diluting particulate deposits:

(1) For long-term sampling at a site, test samples should be returned to the laboratory by express mail service, or air express service or by being hand-carried, and should then be analyzed by scanning electron microscopy.

(2) The estimated particulate loading (deposit is barely visible to the naked eye) should be bracketed by varying the filtration rate and using the same time, or by varying the time and using the same filtration rate.

(3) An automatic particle counter, such as a light-scattering instrument (0.3-μm detection) or a real-time mass monitor (0.1-μm detection), should be used to obtain an approximate particulate-loading level of the area.

Although any one of the three techniques will work, the suggested technique is to take the samples as a set, varying the sampling rates and using the same time so as to obtain filter samples with different particulate loadings. Each set is composed of a minimum of four 37-mm-diameter or 47-mm-diameter filter units--three for different particulate loadings (low, medium, high), and the fourth for a field blank. Suggested sampling rates are 0 for the field blank, 2.48 l/min for the low loading, 7.45 l/m for the medium, and 17.62 l/min for the high, for a 30 min sampling period using a 47-mm-diameter filter holder. Simultaneous sampling will provide at least one sample with a particulate loading suitable for direct EM analysis.

TSP's range from 10 $\mu g/m^3$ in remote, nonurban areas, to 60 $\mu g/m^3$ in near-urban areas, to 220 $\mu g/m^3$ in urban areas. However, for heavily polluted areas, TSP levels may reach 2000 $\mu g/m^3$. A loading of 5 to 10 μg per cm^2 of filter is adequate for EM analysis; values beyond 20 to 25 μg per cm^2 require a dilution treatment. As an example, for 47-mm-diameter filters at face velocities of 3.0 cm/s (2.48 l/min), 9.0 cm/s (7.45 l/min), and 21.2 cm/s

(17.62 l/min), respectively, air volumes of 74.4 l, 223.5 l, and 528.6 l are sampled in 30 min. For a TSP level of 200 $\mu g/m^3$, 14.88 µg (1.07 $\mu g/cm^2$), 44.7 µg (3.23 $\mu g/cm^2$), and 105.7 µg (7.63 $\mu g/cm^2$), respectively, would be collected on 47-mm-diameter filters (which would have effective filtration areas of 13.85 cm^2). The sampling time could be increased to 60 min for areas having lower TSP levels, or reduced in a heavily polluted area (source emissions).

Airborne samples from emission sources contain coarse particles (above the respirable size) of large matrix structures, binder materials, road dust, clay minerals, fillers, and other materials. For these samples, a fifth filter unit can be added that has a size-selective inlet (cyclone, impactor, or elutriator) attached prior to the filter unit. The flow pattern and flow rates of the tandem sampling arrangement must be checked before use. A satisfactory, tested combination presently used in California is a cyclone-filter unit with a D_{50} cut-off of 2.5 µm at 21.7 l/min, and a D_{50} cut-off of 3.5 µm at 15.4 l/min (John and Reischl, 1980). Additional sampling devices, such as impingers (used in biological sampling), impactors, and other designated filter units (for TSP, XRD, or x-ray fluorescence (XRF), for example) can be added to the system to obtain supplementary as well as interrelated data.

This expandable multifilter sampling unit, designated Hydra, offers the following advantages:

(1) It is small, inexpensive, and compact, so that an adult can easily handle it.

(2) It is efficiently designed, and includes a tripod, sampling pump, manifold, critical orifices, and a row of preloaded 37-mm-diameter or 47-mm-diameter filter holders. A rain/wind shield, size-selective cyclone-filter units, tubing, and other extras can be added as needed.

(3) Its sample preparation steps and handling are minimized.

(4) It allows complementary as well as supplementary analysis (TSP, size fractionation, bacteria, and XRF, for example), although additional air sampling capacity is required.

(5) It accommodates ambient air and source emission samples, with or without a size-selective inlet.

(6) It allows synchronous sampling in several places in the vicinity following the same sampling procedure, thereby accommodating particulate concentration fluctuations.

(7) It includes filter holders that serve as transport and storage units.

Hydra's disadvantages are a short sampling period, which may catch an episode; a small sampling quantity or volume, which may not indicate the presence of asbestos fibers; and a detection limit of 2×10^4 fibers/m^3 for sampling 1 m^3 of air with the 47-mm-diameter filter.

Using 8 inch x 10 inch, or 102-mm-diameter filter sizes, is not recommended. The sampling units are designed for purposes other than microscopy. Interchanging the type of sample substrate filter (glass fiber or paper to polycarbonate) does not correct the inherent problems of filter size and sampling unit.

Sample Storage and Transport--

Once the sample is acquired, its integrity must be assured, and contamination and loss of fibers prevented, until it is examined under the EM. The low cost and small size of the 37-mm-diameter and 47-mm-diameter filter holders enables them to be used as combination storage and transport containers. The filter holders should be maintained in a horizontal position during storage and transport to the laboratory so that the particulate-loaded filters can be removed under optimally controlled conditions in the laboratory.

For 47-mm-diameter holders (open-face) to be used in transport or storage, the screw cap is carefully removed, and the shiny, waxy, stiff separator paper used to keep the polycarbonate filters apart is carefully placed on the retaining ring. The cap is then carefully screwed back on so that the separator paper seals and protects the particulate-loaded filter without touching it. The 37-mm-diameter, three-piece filter holder (aerosol monitor) is used in its open-face position, and capped after usage for transport and storage.

When the more expensive 47-mm-diameter holder is to be re-used immediately, the particulate-loaded filter should be carefully removed and placed in a 47-mm-diameter Petri-slide (such as that manufactured by the Millipore Corp.*) This transfer takes place in the field rather than in the laboratory, so that the Petri-slide should be taken into the field. The 37-mm-diameter filter holder or the 47-mm-diameter holder/Petri-slide should be secured and all necessary sample identification marks and symbols applied to the holder.

3. Sample Preparation for Analysis--Grid Transfer

Carbon-Coating the Filter--

The polycarbonate filter, with the sample deposit and suitable blanks, should be coated with carbon as soon as possible after sampling is completed. To begin this procedure, the particulate-loaded 47-mm-diameter polycarbonate filter is removed from the holder and transferred carefully to an open-faced 47-mm-diameter Petri-slide for carbon-coating in the vacuum evaporator (see Figure A1, Appendix A). If the 47-mm-diameter filter is already in the Petri-slide, the cover is replaced with an open-face cover, minimizing filter disruption. The 37-mm-diameter filter is left in the holder, but the upper lid is removed to create an open-faced filter. The open-faced holders are placed on the rotating turntable in the vacuum

* Millipore Corp., 80-T Ashby Rd., Bedford, Mass. 01730

evaporator for carbon-coating. Figure A2 shows the multiple-coating arrangement in the evaporator; Figure A3 shows a close-up of the 37-mm-diameter and the modified 47-mm-diameter holders for carbon-coating.

For archival filters and those of larger sizes, portions of about 2.5 cm x 2.5 cm should be cut midway between the center and edge using a scalpel. The portions are then attached with cellophane tape to a clean glass microscope slide and placed on the turntable in the vacuum evaporator for coating.

Any high-vacuum carbon evaporator may be used to carbon-coat the filters (CAUTION: carbon sputtering devices should not be used). Typically, the electrodes are adjusted to a height of 10 cm above the level of the filters. A spectrographically pure carbon electrode sharpened to a neck of 0.1 cm x 0.5 cm is used as the evaporating electrode. The sharpened electrode is placed in its spring-loaded holder so that the neck rests against the flat surface of a second carbon electrode.

The manufacturer's instructions should be followed to obtain a vacuum of about 1.33×10^{-3} Pa (1×10^{-5} torr) in the bell jar of the evaporator. With the turntable in motion, the neck of the carbon electrode is evaporated by increasing the electrode current to about 15 A in 10 s, followed by 20 to 25 A for 25 to 30 s. If the turntable is not used during carbon evaporation, the particulate matter may not be coated from all sides, resulting in an undesirable shadowing effect. The evaporation should proceed in a series of short bursts until the neck of the electrode is consumed. Continuous prolonged evaporation should be avoided, since overheating and consequent degradation of the polycarbonate filter may occur, impeding the subsequent step of dissolving the filter. The evaporation process may be observed by viewing the arc through welders goggles (CAUTION: never look at the arc without appropriate eye protection). Preliminary calculations show that a carbon neck of 5 mm^3 volume, when evaporated over a spherical surface 10 cm in radius, will yield a carbon layer that is 40 nm thick.

Following carbon-coating, the vacuum chamber is slowly returned to ambient pressure, and the filters are removed and placed in their respective holders or in clean, marked Petri dishes for storage on a clean bench.

Transfer of the Sample to the EM Grid--

Transferring the collected particulates from the carbon-coated polycarbonate filter to an EM grid is accomplished in a clean room or on a class-100 clean bench. The transfer is made in a Jaffe wick washer, which is usually a glass Petri dish containing a substrate to support the EM grid/carbon-coated membrane filter combination. Solvent is added to a level to just wet the combination and cause gentle dissolution of the membrane with minimum loss or dislocation of the particulates, resulting in a membrane-free EM grid with particles embedded in the carbon film coating. The substrate support can be stainless steel mesh bridges, filter papers, urethane foams, or combinations of these.

The refined Jaffe wick washer is described as follows:

(1) The glass Petri dish (diameter, 10 cm; height, 1.5 cm) is made airtight by grinding the top edge of the bottom dish with the bottom of the cover dish, with water and carborundum* powder (80 mesh); this creates a ground-glass seal (closer fit) and minimizes the need to refill the Petri dish with added solvent. (The usual glass Petri dish was found not to retain the solvent for long periods of time, and unless the wicking substrate is kept continuously wet, poor solubility of the membrane filter results, leading to a poor-quality EM grid).

(2) A combination of foam and a single sheet of 9-cm filter paper is used as the substrate support. A 3-cm x 3-cm x 0.6-cm piece of polyurethane foam (the packing in Polaroid film boxes) is cut and placed in the bottom dish. A 0.5-inch V-shaped notch is cut into the filter paper; the notch is oriented in line with the side of the foam, creating a well for adding solvent. Spectrographic-grade chloroform (solvent) is poured into the Petri dish through the notch until it is level with the top of the foam (also level with the paper). The foam will swell, and care is needed to avoid adding solvent above the filter paper.

(3) On top of the filter paper, pieces of 100-mesh stainless steel screen (0.6 cm x 0.6 cm) are placed, usually in two rows, to make several grid transfers at one time (for such uses as replicas), and to facilitate maintenance of proper identity of each transfer.

(4) A 3-mm section (usually midway between the center and edge) of the carbon-coated polycarbonate filter is cut in a rocking motion with a scalpel. The section may be a square, rectangle, or triangle, and should just cover the 3-mm EM grid.

(5) A section is laid carbon-side down on a 200-mesh carbon-coated EM grid. (Alternatively, Formvar-coated† grids or uncoated EM grids may be used. Here, the carbon coating on the polycarbonate filter forms the grid substrate.) Minor overlap or underlap of the grid by the filter section can be tolerated, since only the central 2-mm portion of the grid is scanned in the microscope. The EM grid and filter combination is picked up at the edges with the tweezers and carefully laid on the damp 100-mesh stainless steel screen. The EM grid-filter combination will immediately "wet out" and remain on the screen.

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* Carborundum is a registered trademark of the Carborundum Co., Carborundum Center, Niagara Falls, N.Y. 14302.

† Formvar is a registered trademark of the Monsanto Company, 800 N. Lindbergh Blvd., St. Louis, Mo.

(6) Once all specimens are placed in the washer, more solvent is carefully added through the notch to maintain the liquid level so that it just touches the top of the paper filter. Raising the solvent level any higher may float the EM grid off the mesh or displace the polycarbonate filter section.

(7) The cover is placed in the washer and oriented in place over the specimen, and a map of the filter/grid/screen arrangement is made on the glass cover and in the logbook.

(8) Solvent (chloroform) is added periodically to maintain the level within the washer until the filter is completely dissolved by the wicking action (24 to 48 h).

(9) The temperature in the room must remain relatively constant to minimize condensation of solvent on the bottom of the cover and subsequent falling of solvent drops on the EM grid. Should day-night or other temperature differentials occur, solvent condensation on the under-surface of the cover can be minimized by placing the Jaffe washer at a slight tilt (three glass slides under one edge of the Petri dish parallel to the row of grids) to allow the condensation drops to flow toward the lower edge rather than fall on the EM grids. At temperatures lower than 20°C (68°F), the complete filter solution may take longer than 72 h.

(10) After the polymer is completely dissolved, the stainless steel mesh screen with the EM grid is picked up while wet and set on lens paper tacked to the bottom of a separate Petri dish. The EM grid is then lifted from and placed next to the screen to dry. When all traces of solvent have evaporated, the grid is stored in a grid box and identified by location and grid box in the logbook.

Figure A4 illustrates the Jaffe wick washer method; Figure A5 shows the washer. The foam/filter combination is presently preferred, as is use of a closely fitted (by means of the ground-glass seal) Petri dish.

Gold Coating—

An additional step will aid in subjectively evaluating the SAED pattern. This step is required for specimens from the upper Great Lakes area and for those of unknown origins. After the particulates on the filter are transferred to the EM grid, the grid is held to a glass slide with double-stick tape for gold-coating in the vacuum evaporator. Several EM grids may be taped to the glass slide with double-stick tape for gold-coating in the vacuum evaporator. For comparison, one-half of the EM grids may be coated and the other one-half not coated; recognition of the gold-coating is helpful in searching and x-ray analysis. Several EM grids may be taped to the glass slide for coating at one time. Approximately 10 mm of 0.015-cm-diameter

(0.006-inch) pure gold wire is placed in a tungsten basket (10 cm from the rotating table holding the EM grids) and evaporated onto the grid.

The thin gold-coating establishes an internal standard for SAED analysis. For some mineral species, an internal standard will clarify visual identification of the pattern of a fibrous particulate as being or not being an amphibole species (for example, Minnesotaite as opposed to Amosite). With experience, differentiation in SAED patterns can be observed. For samples of known geographic origins, gold-coating is optional, since the additional coating hinders observation and identification of small-diameter chrysotile fibers.

4. TEM Examination and Data Collection

Figure A10 shows a modern TEM with capabilities for elemental analysis with an EDS. The grid is observed in the TEM at magnifications of 250X and 1000X to determine its suitability for detailed study at higher magnification. The grid is rejected and a new grid used if: (1) the carbon film over a majority of the grid openings is damaged and not intact; (2) the specimen is dark due to incomplete dissolution of the polycarbonate filter; or (3) the particulate loading is too light (unless a blank) or too heavy with particle-particle interactions or overlaps.

TEM Analysis (Morphology, SAED, and X-Ray Analysis)—

The following guidelines are observed for consistency in the analytical protocol:

(1) Magnification at the fluorescent screen is determined by calibration with a diffraction-grating replica in the specimen holder.

(2) A field of view or "gate" is defined. On some microscopes, the central rectangular portion of the fluorescent screen, which is lifted for photographic purposes, is convenient to use. On others, a scribed circle or the entire circular screen may be used as the field of view. The area of the field of view must be accurately measurable.

(3) The grid opening is selected on a random basis.

(4) The analysis, morphology, and SAED are performed at a tilt angle of 0°.

(5) The recommended instrument settings are: accelerating voltage, 100 kV; beam current, 100 μA; film magnification, 20,000X (which is equivalent to 16,000X on the fluorescent screen for this instrument); and concentric circles of radii 1, 2, 3, and 4 cm on the fluorescent screen.

(6) The grid opening is measured at low magnification (about 1000X).

(7) Since asbestos fibers are found isolated as well as with each other or with other particles in varying arrangements, the fibrous particulates are characterized as asbestos structures:

Fiber (F) is a particle with an aspect ratio of 3:1 or greater with substantially parallel sides.

Bundle (B) is a particulate composed of fibers in a parallel arrangement, with each fiber closer than the diameter of one fiber.

Cluster (Cl) is a particulate with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group.

Matrix is a fiber or fibers with one end free and the other end embedded or hidden by a particulate.

Combinations of structures, such as matrix and cluster, matrix and bundle, or bundle and cluster, are categorized by the dominant fiber quality--cluster, bundle, and matrix.

(8) Counting rules for single fibers, which are illustrated in Figure A7 are as follows:

(a) Particulates meeting the definition of fiber are isolated by themselves. With this definition, edge view of flakes, fragments from cleavage planes, and scrolls, for example, may be counted as fibers.

(b) Count as single entities if separation is equal to or greater than the diameter of a single fiber.

(c) Count as single entities if three ends can be seen.

(d) Count as single entities if four ends can be seen.

(e) In general, fibers that touch or cross are counted separately.

(f) Two or more fibers are counted as a bundle if the distances between fibers are less than the diameter of a single fiber, or if the ends cannot be resolved.

(g) Fibrils attached longitudinally to a fiber are counted as part of the fiber and the size (width) is estimated based on the fiber-to-fibril relationship.

(h) A fiber partially hidden by grid wires (one or two sides of the grid opening) is counted, but labeled as an X-fiber (X-F) in the structure column. If the number of X-fibers is high enough to affect the size distribution (mass, etc.), a large-mesh EM grid should be used, such as 100 mesh (about 200 μm wide).

(9) Sizing rules for asbestos structures are:

(a) For fibers, widths and lengths are obtained by orienting the fibers to the inscribed circles on the fluorescent screen. Since estimates are within ±1 mm, small-diameter fibers have greater margins of error. Fibers less than 1 mm at the fluorescent screen magnification level are characterized as being 1 mm. A cylindrical shape is assumed for fibers. X-fibers are sized by measuring their entire visible portions in the grid opening.

(b) Bundles and clusters are sized by estimating their widths and lengths. The sum of individual diameters is used to obtain the total width, and an average length for the total length. A laminar-sheet shape is assumed, with the average diameter of the individual fiber as the thickness.

(c) Matrices are sized by adding the best estimates of individual fiber components. A laminar or sheet structure is assumed for volume calculation.

(10) The method of sizing is as follows:

(a) An asbestos structure is recognized, and its location in the rectangular "gate" relative to the sides, inscribed circles, and other particulates is memorized.

(b) The structure is moved to the center for SAED observation and sizing.

(c) Sizing is performed using the inscribed circles. If the structure, such as a fiber, extends beyond the rectangular gate (field of view), it is superimposed across the series of concentric circles (several times, if necessary) until the entire structure is measured.

(d) The structure is returned to its original location by recall of the location, and scanning is continued.

Analytical Procedure--

The analytical procedure is as follows:

(1) EM grid quality is assessed at 250X.

(2) Particulate loading is assessed at 1000X.

(3) A grid opening is selected at random, examined at 1000X, and sized.

(4) A series of parallel traverses is made across the grid opening at the film magnification of 20,000X. Starting at one corner, and using the tilting section of the fluorescent screen as a "gate" or "chute," the grid opening is traversed. Movement through the "gate" is not continuous, but rather is a stop/go motion. On reaching the end of one traverse, the image is moved the width of one "gate," and the traverse is reversed. These parallel traverses are made until the entire grid opening has been scanned.

(5) Asbestos structures are identified morphologically and counted as they enter the "gate."

(6) The asbestos structure is categorized as fiber (with or without X-) bundle, cluster, or matrix, and sized through use of the inscribed circles.

(7) The structure (individual fiber portion) is centered and focused, and the SAED pattern is obtained through use of the field-limiting aperture.

(a) SAED patterns from single fibers of asbestos minerals fall into distinct groups. The chrysotile asbestos pattern has characteristic streaks on layer lines other than the central line, and some streaking also on the central line. Spots of normal sharpness are present on the central layer line and on alternate lines (that is, 2nd, 4th etc.) The repeat distance between layer lines is about 0.53 nm.

(b) Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and have repeat distances between layer lines also of about 0.53 nm. Streaking in layer lines is occasionally present due to crystal structure defects.

(c) Transmission electron micrographs and SAED patterns obtained with asbestos standard samples should be used as guides to fiber identification. An example is the "Asbestos Fiber Atlas" (Mueller et al., 1975).

(8) From visual examination of the SAED pattern, the structure is classified as belonging to one of four categories: (1) chrysotile, (2) amphibole group (includes amosite, crocidolite, anthophyllite, tremolite, and actinolite), (3) ambiguous (incomplete spot patterns), or (4) no identification. SAED patterns cannot be inspected for some fibers. Reasons for the absence of a recognizable diffraction pattern include contamination of the fiber, interference from nearby particles, fibers that are too small or too thick, and nonsuitable orientation of the fiber. Some chrysotile fibers are destroyed in the electron beam, resulting in patterns that fade away within seconds of being formed. Some patterns are very faint and can be seen only under the binocular microscope. In general, the shortest available camera length must be used, and the objective lens current may need to be adjusted to give optimum pattern visibility for correct identification. A 20-cm camera length and a 10X binocular are recommended for inspecting the SAED pattern on the tilted screen.

(9) The specimen holder is tilted for optimum x-ray detection (40° tilt for the JEOL* 100C instrument's Tracor Northern† NS 880 analyzer and Kevex†† detector). The categorized asbestos structure is maintained in its centered position for x-ray analysis by means of the Z-control.

(10) The spot size of the electron beam is reduced and stigmated to overlap the fiber. As an option for STEM instruments, the electron beam may be used in the spot mode and the x-ray analysis performed on a small area of the structure.

(11) The EDS is used to obtain a spectrum of the x-rays generated by the asbestos structure.

(12) The profile of the spectrum is compared with profiles obtained from asbestos standards; the best (closest) match identifies and categorizes the structure. The image of the spectrum may be photographed, or the peak heights (Na, Mg, Si, Ca, Fe) recorded for normalizing at a later time. No background spectra or constant acquisition time is required since the shape of the spectrum (profile) is the criteria. Acquisition of x-ray counts may be to a constant time; to a constant peak height for a selected element, such as silicon (1.74 keV); or just

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* JEOL (U.S.A.) Inc., 11 Dearborn Road, Peabody, Mass. 01960

† Tracor Northern Inc., 2551-T.W. Beltway Hwy., Middleton, Wis. 53562

†† Kevex Corp., Chess Dr., Foster City, Calif. 94404

long enough to get an adequate idea of the profile of the spectra, and then aborted. Figure A11 illustrates spectra obtained from various asbestos standards and used as referenced profiles.

(13) The specimen holder is returned to 0° tilt to examine other asbestos structures.

(14) Scanning is continued until all structures are identified, measured, analyzed, and categorized in the grid opening.

(15) Additional grid openings are selected, scanned, and counted until either the total number of structures counted exceeds 100 per known area, or a minimum of 10 grid openings has been scanned, whichever is first.

(16) The TEM data should be recorded in a systematic form so that they can be processed rapidly. Sample information, instrument parameters, and the sequence of operations should be tabulated for ease in data reduction and subsequent reporting of results. Figure A12 shows an example of a data sheet used in Level II analysis.

Figure A9 illustrates the method of scanning a full-grid opening. The "field of view" method of counting, which is based on randomly selected fields of view, has been discontinued. Originally, the method was recommended for medium loading level on the filter (50 to 300 fibers per grid opening). However, if samples are collected at three different loading levels and the optimum is selected, this medium loading on the filter will not be used. Samples with grid openings containing 50 to 300 fibers may be used as laboratory fiber preparations or selected source samples, but in field samples, the particulate loading is usually of much higher concentration than the fiber. Filter loading is characterized by particulate concentration, not by fiber concentration.

EDS is relatively time-consuming, and becomes redundant if used as repetitive analysis for a confirmatory check on chrysotile fibers. Chrysotile identity by morphology and visual SAED analysis is not as controversial as amphibole identification and categorization.

The following rules are recommended for EDS analysis (Level II):

(1) For chrysotile structure identification, the first five are analyzed by EDS, then one out of every 10.

(2) For amphibole structure identification, the first 10 are analyzed by EDS, then one out of every 10.

(3) For amphibole structure identification and categorization, all confirmed amphiboles are analyzed by EDS.

(4) For ambiguous structure identification and categorization, all are analyzed by EDS.

Energy dispersive x-ray analysis as used in asbestos analysis is semiquantitative at best. X-ray analyzer manufacturers may claim quantitative results based on calibration standards and sophisticated computer software, but such claims are based on stoichiometric materials and extension of work with XRF instrumentation. Asbestos has a varying elemental composition. The electron beam in an EM is of varying size, and not all instruments are equipped to measure the beam current hitting the specimen. The size of the specimen has an effect on the X-ray output, and nearby materials may fluoresce and add to the overall x-ray signals being generated. Moreover, specimen tilting results in a loss of x-ray acquisition from particles hidden by grid wires or by other particles.

The only consistency in x-ray analysis is that the intensity of the output, within restrictions, is proportional to the mass, therefore providing the semiquantitative analytical possibility. Asbestos minerals have been found to have a characteristic profile, although not an exact duplicate of each other. For example, the Mg:Si ratio of chrysotile may vary from 5:10 to 10:10, averaging about 7:10. The ratio can be used to confirm the morphology and visual SAED analysis.

Table 1 illustrates the phenomena of variability with resemblance for some of the amphibole fibers. Peak heights and profile measurements were taken.

To aid in the visual perspective of the spectrum profile, the peak heights were normalized to a silicon value of 10, resulting in a five-number series that is relatively easy to visualize--as in the following examples:

chrysotile	~ 0-7-10-0-0
tremolite	~ 0-4-10-3-<1
crocidolite	~ 1-1-10-0-6
anthophyllite	~ 0-3-10-0-1
amosite	~ 0-2-10-0-7

These relationships are approximate, since chrysotile can vary from 0-5-10-0-0 to 0-10-10-0-0. However, for the others, the variation is only about one point, such that the profile (shape) of the five elements (Na, Mg, Si, Ca, Fe) is recognizable.

5. Data Reduction and Reporting of Results

Data Reduction—

From the data sheet, size measurements are converted to microns (16,000X screen magnification), mass of asbestos structure is calculated, and other characterizing parameters are calculated through use of a hand calculator or computer. (Appendix C, an example of a computer printout from Level II analysis, shows reduced data--that is, what was found on the specified number of grid openings or area examined.) These measurements are summarized and related to the volume of air sampled and the total effective filtration area

TABLE 1. PROFILE COMPARISON OF ASBESTOS STANDARDS

Asbestos Type	Size, μ	Na	Mg	Si	Ca	Fe	Profile
Amosite (GF-38A)	0.19 x 1.44 (stigmate)		182	497		346	0-4-10-0-8
	0.19 x 0.75 (STEM)		186	528		387	0-4-10-0-7
	0.19 x 1.25		181	352		289	0-5-10-0-8
	0.19 x 0.88 (100 s)		226	870		674	0-3-10-0-8
	0.25 x 1.81 (100 s)		576	4207		3338	0-1-10-0-8
	0.12 x 1.56		253	2049		1515	0-1-10-0-7
	0.31 x 2.38		256	2127		1613	0-1-10-0-8
	0.19 x 1.56		276	1696		1116	0-2-10-0-7
	Repeat		477	2945		1959	0-2-10-0-7
Anthophyllite (AF-45)	0.56 x 2.38 (stigmate)		631	2577		349	0-2-10-0-1
	0.31 x 2.38 (stigmate)		640	1670		71	0-4-10-0-0
	0.31 x 5.19 (stigmate)		1064	3610		466	0-3-10-0-1
	0.19 x 1.56 (stigmate)		507	2191		309	0-2-10-0-1
	0.19 x 1.88 (stigmate)		787	2286		257	0-3-10-0-1
Crocidolite (CR-37)	0.19 x 0.81 (stigmate)	131	100	885		501	2-1-10-0-6
	0.06 x 0.50 (stigmate)	28	28	205		115	1-1-10-0-6
	0.06 x 0.69 (stigmate)	37	35	171		96	2-2-10-0-6
	0.12 x 1.00 (stigmate)	44	53	379		204	1-1-10-0-5
	Repeat (STEM)	70	64	612		333	1-1-10-0-5
	0.12 x 0.62 (stigmate)	56	65	479		260	1-1-10-0-5
	0.12 x 1.12 (stigmate)	53	56	326		166	2-2-10-0-5
	0.19 x 1.56 (stigmate)	78	83	735		421	1-1-10-0-6
	0.06 x 1.69 (stigmate)	45	48	290		159	2-2-10-0-6
	Repeat (STEM)	72	85	892		463	1-1-10-0-5
	Repeat (STEM)	35	42	373		237	1-1-10-0-6
	Repeat (STEM)	16	22	166		104	1-1-10-0-6
Tremolite (T-79)	0.38 x 2.19 (stigmate)		138	368	93		0-4-10-2-0
	0.38 x 2.19 (spot)		114	327	80		0-4-10-2-0
	0.25 x 1.75 (stigmate)		80	197	65		0-4-10-3-0
	0.25 x 1.75 (spot)		95	252	62		0-4-10-2-0
	Repeat (stigmate)		70	211	51		0-3-10-2-0
	(STEM-100 s)		376	1118	245		1-3-10-2-0
	(STEM-100 s)		135	364	72		0-4-10-2-0
	(STEM-100 s)		1454	4810	1235		0-3-10-3-0
	(STEM-100 s)		64	191	48		0-3-10-2-0
	(STEM-100 s)		1072	3114	882		0-3-10-3-0
	(STEM-40 s)		46	113	27		0-4-10-2-0
	(STEM-40 s)		123	333	94		0-4-10-3-0

(area of deposit). Size measurements of X-fibers may be doubled and noted, or kept as a separate category.

Fiber number concentration is calculated from the equation

$$\text{Fibers/m}^3 = \frac{\text{Total no. of fibers}}{\text{No. of EM fields}} \times \frac{\text{Total effective filter area, cm}^2}{\text{Area of an EM field, cm}^2} \times \frac{1}{\text{Volume of air sampled, m}^3}$$

The number of X-fibers, bundles, clusters, and matrices are calculated in a similar manner. X-fibers may be included with fibers if they are few in number. Similarly, their corresponding mass (from their size measurements) may be included.

Fiber mass for each type of asbestos (chrysotile or amphibole) in the sample is calculated by assuming that both chrysotiles and amphiboles have circular cross-sections (cylindrical shape) and that the width measurements are one diameter. The density of chrysotile is assumed to be 2.6 g/cm^3, and of amphiboles to be 3.0 g/cm^3. The individual mass is calculated from the equation

$$\text{Mass, } \mu g = \frac{\pi}{4} \times (\text{length, } \mu m) \times (\text{diameter, } \mu m)^2 \times (\text{density, } g/cm^3) \times 10^{-6}$$

The total mass concentration of fibers for each type of asbestos is then calculated from the total mass of all the individual fibers of that type.

The individual masses of bundles, clusters, and matrices are calculated by assuming a laminar or sheet-like structure with an average thickness of the fiber make-up of the structure. Again, the density of chrysotile is assumed to be 2.6 g/cm^3, and of amphiboles to be 3.0 g/cm^3. The individual masses are calculated from the equation

$$\text{Mass, } \mu g = (\text{length, } \mu m) \times (\text{width, } \mu m) \times (\text{thickness, } \mu m) \times (\text{density, } g/cm^3) \times 10^{-6}$$

The total mass for each type of structure for each type of asbestos is the sum of all the individual masses.

Other characterizing parameters of the asbestos structures are: (1) length and width distribution of fibers, (2) aspect ratio distribution of fibers, and (3) relationships of fibers, bundles, clusters, and matrices.

Reporting of Results—

The data and their subsequent reduction are reported as summarized, or can be further reduced to present the interrelationships of the various characterizing parameters. Figure A13 is an example of the EM data report; Figure A14 is an example of the sample summary report.

The methodology can establish the limits of identity for unknown samples, act as a QC/QA method for Level I analysis, and satisfy most of the identification criteria for asbestos.

6. Quality Control/Quality Assurance

Sampling procedures will vary depending on the type of sample, objectives of the sampling, and time/cost factors. The primary goals of sampling are to obtain a representative sample at the location and time of sampling, and to maintain sample integrity. The sampling team will have written sampling procedures, and the field chief and/or designated individual will be responsible for all record-keeping (including sample identification, labeling, logging of data, site description, and meteorological conditions), pre- and post-collection checks, and continuous sample custody and sign-outs until the sample is delivered to the laboratory and transferred to the appropriate quality assurance officer (QAO). Verification of sampling times, flow rates, equipment calibration, and taking of field blanks will be checked and recorded in the field logbook.

Samples are turned over to the QAO for logging into a project logbook. Each sample is carefully examined for gross features, such as tears, breaks, and overall condition of container. The QAO registers the as-received sample number and other designated information, and assigns a simple internal code number that will accompany the sample through the preparation stage, grid transfer, grid analysis, data reduction, and reporting of results.

After being logged into the project logbook, the sample is transferred to the custody of the electron microscopy staff, where every precaution is taken to maintain sample integrity and to prevent contamination and loss of collected particulates. During storage and transport, the filters in their respective holders are maintained in a horizontal position at all times.

The sample logging, handling, and storing procedures ensure that all samples can be readily located and identified throughout the course of a program. The QAO has divisional responsibility for QC/QA activities, and must see that the laboratory maintains high standards. He must be aware of current standards of analysis, and must ensure that internal quality control standards, instrument calibration, and records of samples and completed analyses are kept for ease of later retrieval and use.

For quality control, internal laboratory blanks are analyzed at least once a week, which may or may not coincide with a sample batch blank. In addition, a magnification calibration of the EM using a carbon grating replica (2,160 lines per mm) is performed once a week. The results are recorded in an EM instrument log, along with other routine instrumental performance checks. All photographs, TEM, SEM, and STEM images are recorded in a photo log. These QC results are documented for inspection by the QAO.

IIT RESEARCH INSTITUTE STRUCTURE ANALYSIS DATA REPORT AND SUMMARY TABLE FOR A REPRESENTATIVE SAMPLE

IIT RESEARCH INSTITUTE STRUCTURE ANALYSIS DATA
INDIVIDUAL OBJECT DATA TABLE (F=FIBER, B=BUNDLE, C=CLUSTER, M=MATRIX)
TABLE PREPARATION DATE: 11-OCT-85

SAMPLE CODE: C06610-0120 car-N-31

Grd Opn	Obj	Str	Size (Micron) Depth	Width	Length	Ratio	Mass (Picogram) Chrysotile	Amphibole	Ambig	Not Asbe	No Patt	X-Ray
3	1	F	0.000	0.062	1.19	19.0	.	0.011	.	.	.	
8	2	F	0.000	0.625	5.94	9.5	.	5.464	.	.	.	
9	3	F	0.000	1.250	5.13	4.1	.	.	.	X	.	
11	4	F	0.000	0.250	2.81	11.3	.	0.414	.	.	.	
15	5	F	0.000	0.375	2.75	7.3	.	0.911	.	.	.	
15	6	F	0.000	0.625	4.37	7.0	.	.	.	X	.	
16	7	F	0.000	0.187	1.19	6.3	.	0.098	.	.	.	
17	8	F	0.000	0.250	1.87	7.5	.	0.276	.	.	.	
17	9	F	0.000	0.250	3.50	14.0	.	0.515	.	.	.	
17	10	C	0.187	0.812	5.31	6.5	.	2.428	.	.	.	
17	11	F	0.000	0.187	1.00	5.3	.	0.083	.	.	.	
19	12	F	0.000	0.562	6.87	12.2	.	5.125	.	.	.	
19	13	F	0.000	0.375	5.94	15.8	.	1.967	.	.	.	
			Total Mass (Picogram)=				0.000	17.293				
			Total Count			=	0.	11.	0.	2.	0.	

IIT RESEARCH INSTITUTE STRUCTURE ANALYSIS DATA
SINGLE SAMPLE SUMMARY TABLES
SAMPLE CODE: C06610-0120 TABLE PREPARATION DATE: 11-OCT-85

Aerosol Object Count And Calculated Object Mass Characteristics

Object Structure	Type	Actual Object Count	Number Concen. (Number Per Cu M)	Mass Concen. (Picogram Per Cu M)	Average Width (Micron)	Average Length (Micron)	Average Length To Width Ratio
Fiber	Chrysotile	0.	0.	0.0	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
	Amphibole	10.	8057.	11976.6	0.31 ± 0.17	3.31 ± 2.20	10.83 ± 4.47
	Other	2.	1611.		0.94 ± 0.44	4.75 ± 0.53	5.55 ± 2.05
	All Fiber	12.	9668.		0.42 ± 0.32	3.55 ± 2.07	9.95 ± 4.57
Cluster	Chrysotile	0.	0.	0.0	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
	Amphibole	1.	806.	1956.2	0.81 ± 0.00	5.31 ± 0.00	6.54 ± 0.00
	Other	0.	0.		0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
	All Cluster	1.	806.		0.81 ± 0.00	5.31 ± 0.00	6.54 ± 0.00

Sample Collection and Preparation Data

Air Volume	=	1.00 Cu M
Deposit Area	=	1.00 Sq Cm
Ashed Area	=	1.00 Sq Cm
Redeposit Area	=	1.00 Sq Cm

Grid Data

Grid ID:	MISC #19/C3 + C4	
Individual Grid Opening	=	0.000062 Sq Cm
Number of Grid Openings	=	20
Film Magnification	=	20000

APPENDIX C

STATISTICAL METHOD

The statistical significance of the observed difference between results obtained by PCM and TEM analyses of samples collected under nonaggressive and aggressive conditions and the difference between nonaggressive and aggressive sampling conditions obtained by PCM and TEM analyses was determined through the application of the Mann-Whitney[a] test. The Mann-Whitney test was selected from among several procedures for hypothesis testing because its application does not require any prior knowledge of the underlying probability distribution function of the data. A cursory inspection of the data suggests a positive skewness (i.e., a few values that are very high compared with the other members of a data set). For this reason, the geometric mean, rather than the arithmetic mean, was used as a measure of central tendency.

Mann-Whitney is used to test the null hypothesis that two different samples were taken from the same population. Rejection of the null hypothesis implies that the two samples are from two different populations. The test assumes that sample A consists of m observations and sample B consists of n observations, where $n \leq m$. The data values from the two samples are ranked into a single set with m + n observations. The statistic T is calculated next; this is the sum of the ranks of the values in sample B. Finally, it is necessary to determine the probability that the calculated value of T would differ as much or more from the expected value of T for the m + n observations. Tables of exact probabilities of T are available for small values of m and n. In situations where m and n are not small (as is the case with the data from this study), the normal approximation can be used to calculate the necessary probability. When this probability is small (i.e., less than 0.05), the null hypothesis is rejected.

As an example, consider the data in Table C-1, which shows results obtained by TEM under nonaggressive and aggressive sampling conditions.

The conclusion is that the probability that T would be greater than or equal to 121.5 for a sample of n = 9 when m = 9 is 0.001. This is sufficient cause to reject the null hypothesis that the two sets of data were taken from a single population. On this basis, it is concluded that aggressive sampling yields concentrations of fibers that are much higher than those measured under nonaggressive sampling.

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[a] Mosteller, F., and R. E. K. Rourke. Sturdy Statistics, Nonparametric and Order Statistics, Addison-Wesley. 1973. pp. 54-88.

TABLE C-1. APPLICATION OF THE MANN-WHITNEY TEST TO NONAGGRESSIVE AND AGGRESSIVE RESULTS OBTAINED BY TEM

Nonaggressive		Aggressive	
Value	Rank	Value	Rank
<0.005	1	0.017	8.5
0.006	2.5	0.022	10
0.006	2.5	0.024	11
0.010	4	0.025	12
0.011	5	0.041	14.5
0.012	6	0.041	14.5
0.016	7	0.059	16
0.017	8.5	0.060	17
0.028	13	0.104	18
		T =	121.5

$m = 9$

$n = 9$

$$\mu_T = \frac{n\,(m + n + 1)}{2}$$

$$= \frac{9\,(9 + 9 + 1)}{2}$$

$$= 85.5$$

$$Z = \frac{121.5 - 85.5}{11.32}$$

$$= 3.18$$

$p = 0.001$

$$\sigma^2_T = \frac{n\,m\,(m + n + 1)}{12}$$

$$= \frac{(9)(9)(9 + 9 + 1)}{12}$$

$$= 128.25$$

$$\sigma_T = 11.32$$

APPENDIX D

COMPARISON OF NONAGGRESSIVE AND AGGRESSIVE ASBESTOS SAMPLING RESULTS

Figures D-1 and D-2 show graphical comparisons of sample results taken under nonaggressive and aggressive post-abatement conditions. These figures clearly demonstrate that asbestos fibers and structures measured under aggressive conditions are higher than those measured under nonaggressive conditions.

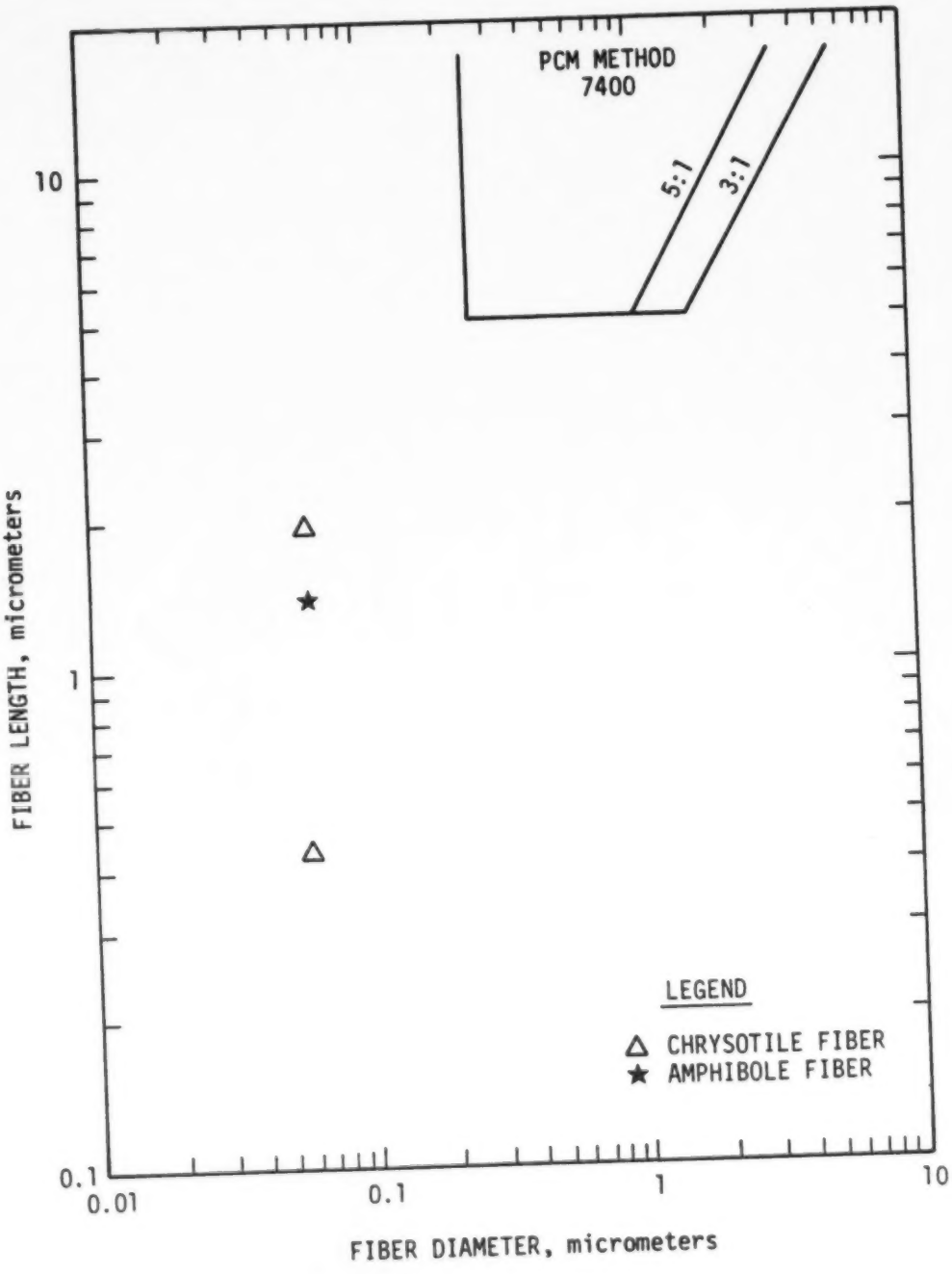

Figure D-1. Plot of fiber length and fiber diameter for a non-aggressive post-abatement air sample in Room 157.

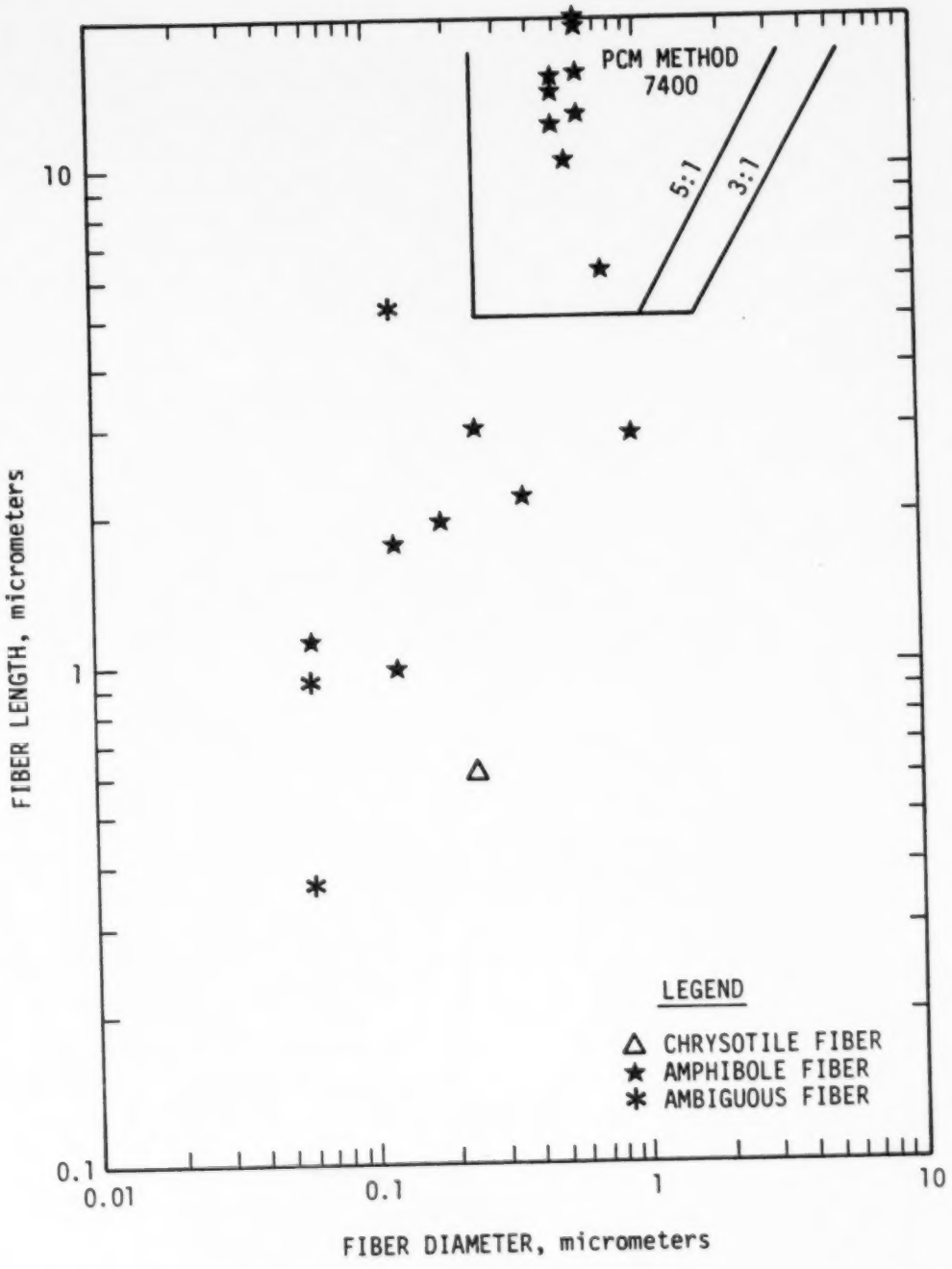

Figure D-2. Plot of fiber length and fiber diameter for an aggressive post-abatement air sample in Room 157.